

# The Chemical Age

VOL LXVI

12 APRIL 1952

No 1709

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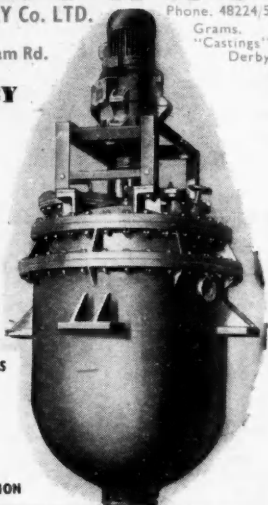
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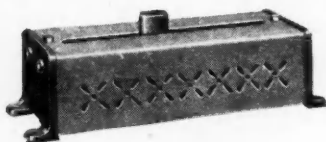
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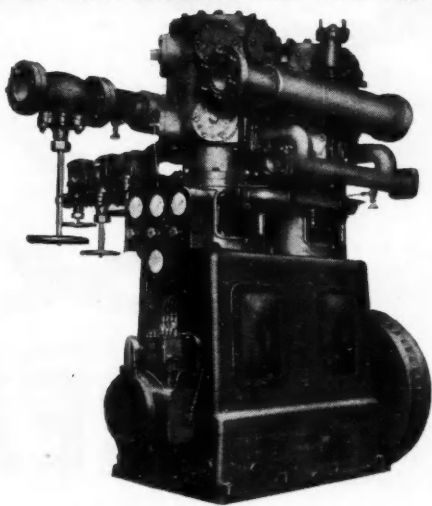


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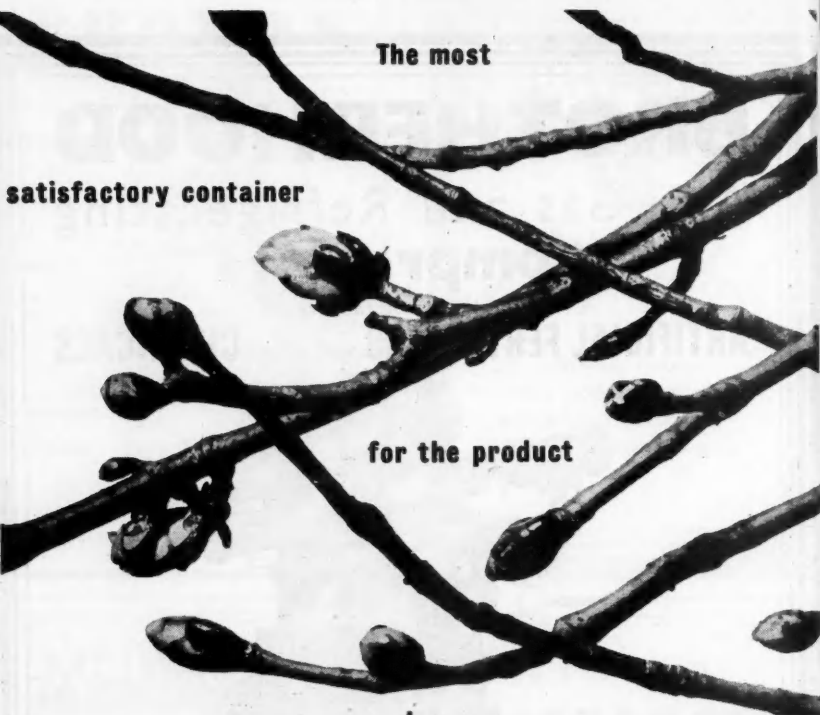
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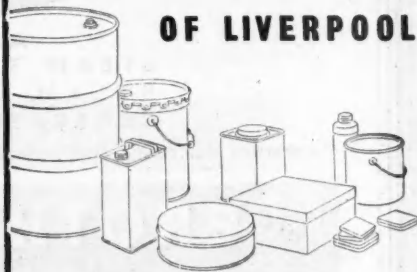
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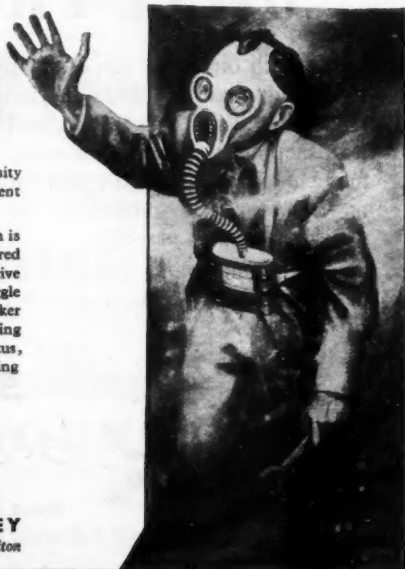
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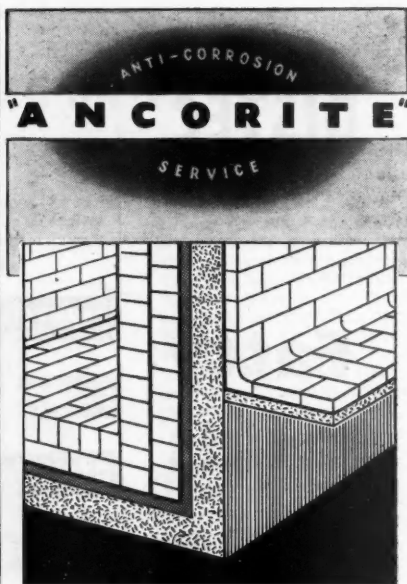
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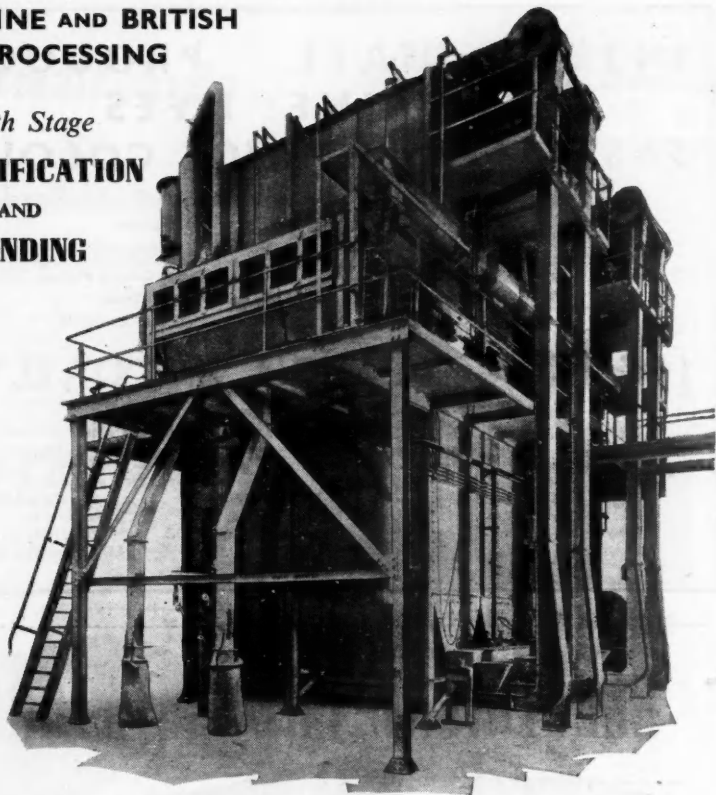
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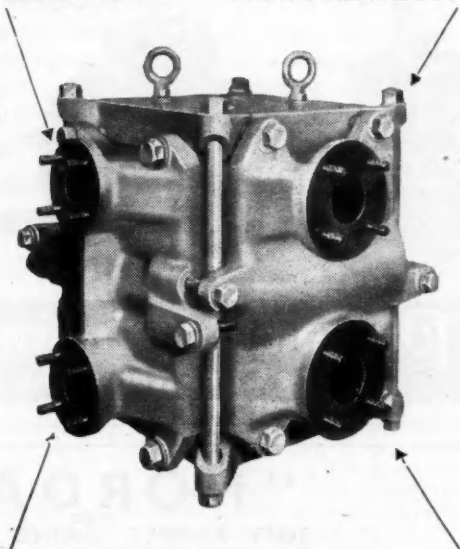


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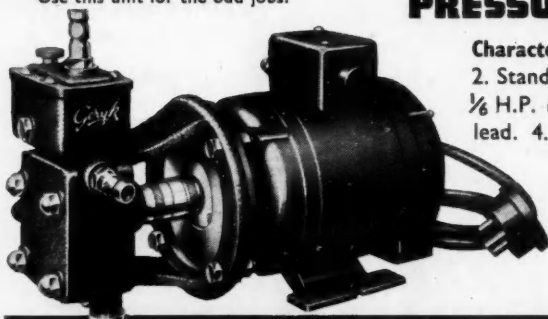
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Volume LXVI

12 April 1952

Number 1709

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## The Look and the Taste

TWO further papers from the SCI conference on the use of chemicals in food have now emerged in print, and they deal respectively (*Chemistry and Industry*, 1952, 11, 238 and 12, 259) with what might be called the aesthetic aspects of food, colour and flavour. It can be argued that neither colour nor flavour has any effect upon the primary function of food, to provide nutrition. Such an argument, however, is coldly inhuman and it certainly ignores psychology. In the kitchen and dining-room, colour is one of the principal properties by which many foods are judged. Colour often indicates freshness or staleness, adequate cooking or over-cooking; and it stimulates appetite. Flavour, even if we ignore the nasal impacts of flavour and consider merely the impacts upon the taste-buds, can decide whether food is eaten at all.

Two basic problems arise from the use of chemical substances for directly contributing colour and flavour to foodstuffs. Is it right to suggest to the consumer, who has no guide other than those of the simple senses, that a food, be it mixed fruit drops, cans of fruit, or tomato sauce, has a more attractive colour than its actual history of treatment provides? No generalised answer seems possible. It is surely necessary to distinguish—at any rate morally—between the totally manu-

factured foodstuff and the natural foodstuff that has merely been processed. Without margarine the fat consumption of much of Europe's population would be disastrously low; but margarine must be given the yellow colour of butter to make most people approach it as a sensible alternative. This does not seem quite the same as artificially adding colouring substances to canned fruit or vegetables to compensate for the loss of colour that often accompanies the canning process. As for flavour additions, generalisation seems even more difficult. Artificial flavourings are almost exclusively used in connection with the very much 'made-up' foods—the fillings of chocolates and other confections, ice cream, drinks like ginger ale, and so on. These foodstuffs do not have natural counterparts and the manufacturer has as much right to make use of flavouring agents to achieve a satisfactory final result as the cook at home. Somewhere a moral line must be drawn and if artificial flavouring is used to imply that natural materials are present when in fact they are not, some protection should be available for the public. The remedy lies in compositional statement.

This, the first problem, can be safely left in the hands of the food industry and existing laws. Bad products in the

guise of better ones do not bring lasting trade; and legislation already and sufficiently sensibly ensures that deliberate fraud is dangerous. Public demand in any case often prefers an artificial to a natural standard of colour. No tomato sauce with only the colour derived from the content of tomatoes would be likely to sell readily by appearance.

The second problem—the question whether the agents used for colour or flavouring are in any way dangerous to health—is quite different. And it is a changing problem, changing rapidly as more and more synthetic substances are developed. Mr. Revie's paper on flavouring agents was devoted to a survey of their gradual development and the question of possible toxicity was raised only in the discussion. It is clear that the specialised industry that produces flavouring essences and substances is highly secretive; this, together with the fact that Mr. Revie stated that to his knowledge the toxicities of synthetic flavouring agents have never been investigated, is certainly disturbing. So long as flavouring essences are derived from natural sources, toxicity risks can be discounted; but it was stated that every new fine chemical (out of 'the infinitely great number of aromatic fine chemicals') is examined to assess its flavour potential.

Future changes in this industry could well produce the problem unequivocally stated in Dr. Peacock's paper on colouring agents. The cancer-disposing properties of certain dyes were never realised until workers in the dye factories provided

statistical and tragic evidence. The carcinogenic potency of a number of dyes is now well-known but 'the synthetic organic chemist today can prepare new substances much faster than they can possibly be tested.' Dr. Peacock urged that the deliberate addition of all synthetic substances to food should be banned. Dr. Peacock approached the problem entirely from the viewpoint of an experimental pathologist whose subject is carcinogenesis; but this should not be looked upon as a discounting bias to his forthright opinion. He is, indeed, specially entitled through his own work to assess the extent of unknown risks that may be taken when entirely new chemicals are brought into contact with living cells.

Perhaps the soundest solution to these modern problems is that adopted by the U.S.A. and Canada and mentioned in Dr. Peacock's paper. There, there is a permitted list of food dyes rather than the prohibited list that operates in this country. One point seems abundantly clear, however. The case for using new synthetic substances for producing colour and flavour effects is very much weaker than the case for using synthetic substances as crop protecting or food preserving agents. In the former use of new chemicals we gain only superficial advantages and there is not a scarcity of either natural products or substances whose long previous use seems to have been quite safe; in the latter we gain very real advantages from higher food production and some new products are far better than their older counterparts.

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## Notes & Comments

### Water, Water, Everywhere . . .

**A**S is well known, a small apparatus for de-salting sea water by ion exchange methods has been developed. When last year the 20-foot boat, *Nova Espero*, left the South Bank Exhibition and sailed for New York, the crew of two relied for the 90-day voyage upon one of the new de-salting kits produced by the Permutit Co., Ltd. Can the conversion of sea water into drinkable water be accomplished on a large scale also? At present, process costs would be too high and in any case ion exchange resins would not handle sufficiently large quantities for sea water to serve as a supplementary source of public water supplies. Considerable research interest is being taken in this problem, however, especially in the United States where several cities have outgrown their natural water supplies. It is stated that Congress may allocate two million dollars to de-salting sea water research though this actually represents a sharp reduction in the 25 million dollars request that was made. Large-scale de-salting seems unlikely to be based upon ion exchange methods alone; these are now experimentally combined with the use of power to dissociation the sodium chloride. The resins are used in sheet or membrane form, not as a bed of granules. A figure for de-salting of 10 to 20 cents per 1,000 gallons has been given by the American Research and Development Corporation as an indication of running costs. The equipment needed would be large and costly and in any place where power was cheap this charge may prove more of a deterrent to development than the likely cost of electricity.

### Leonardo, Scientist

**M**UCH discussion, both verbal and written, has been taking place for some years now on the problem of how far it is possible and desirable to combine a knowledge of the arts with higher technological education. A supreme example of the successful fusion of the artist and scientist is Leonardo da Vinci, of whose birth the 500th anniversary

is being celebrated by an exhibition in the Diploma Galleries of the Royal Academy. While there are many of his finest paintings (including a number lent by Her Majesty the Queen), the East Gallery is devoted to his scientific work and shows 144 drawings and a number of models reconstructing his mechanical inventions. His notebooks, of which more than 5,000 pages are still in existence, are represented by a small but well chosen selection. Leonardo's astonishing originality and ingenuity have been appreciated only fairly recently, for the notebooks were not published until some 70 years ago. It is remarkable, as has been pointed out by Dr. F. Sherwood Taylor, director of the Science Museum, that the only subject Leonardo seems to have been incurious about was chemistry. He may have been repelled by the mysteries and symbols of the alchemists, or maybe felt that it was not a subject suited to his attempts to interpret nature in terms of mechanical structure. Never has so great a scientist had so little influence as Leonardo, for he seems to have had little desire to impart his discoveries. Nevertheless, today when increasing effort is needed to encourage craftsmen in industry, it is good to see that the suggestion made by Sir Robert Hyde (in letters to *The Times*) that apprentices should be encouraged and given the opportunity to visit the exhibition, is meeting with approval.

### Fertiliser Prices Reduced

**A**NEW and rapidly produced Control Price Order removed 12s. 9d. from the ton prices of compound fertilisers, 11s. from the ton price of superphosphate, and 15s. from that of ground phosphate. For those holding stocks of fertilisers the operative date of the change—1 April—must have had additional sting. It was only on the first of February that prices were advanced—on that occasion compounds moved upwards by about £1 per ton according to analysis, superphosphate by 5s. 6d. and ground phosphate by 9s. March has proved to be an active month for the industry as the

mild weather—until the sudden blizzards of the recent period—had brought most farming operations forward. Even if the agricultural year of 1951/52 has been a dull and difficult period for the fertiliser industry, the peak demand during March has not fallen much below that of previous years. The effect of the price reductions, which must fall wholly upon the industry and the trade so far as stocks are concerned, would have been far more serious had the weather in March been less favourable.

### Cost Influences

THE reductions are said to be due to the fall in the price of sulphuric acid and falls in freight costs; both these changes are, of course, of direct concern to costs of rock phosphate and superphosphate manufacture. Another influence tending to ease fertiliser prices is

the fall in jute or hessian bag prices. However, the widespread development of granulation has necessarily converted much of the industry to paper sacks; here, too, price reduction is anticipated in the near future. The cost of packing a ton of fertiliser has swollen more than any other factor in the past ten years and any lasting easement of this accessory cost will be welcomed by farmer and manufacturer alike. No doubt prices will again be reviewed for the 1952/53 farming year which commences on 1 July. It is to be hoped that any further changes—whether downward or upward—will be promptly decided and announced. Frequent alterations in price make production planning almost impossible, and if production cannot be steadily maintained, costs must inevitably rise and this will offset some of the benefits from other cost influences that are now easing.

## New Headquarters

### A Fitting Reward for Growing Activities

A NOTABLE milestone in the progress and development of the Textile Institute was reached on Friday, 4 April, when its new headquarters at 10 Blackfriars Street, Manchester, were officially opened by the president, Mr. G. H. Spencer in the presence of a distinguished gathering representing the ten textile research associations and other textile organisations.

To mark the occasion a well-produced and illustrated brochure has been sent to the Institute's members throughout the world. The Arms of the Institute granted by Letters Patent dated 2 November, 1951, are reproduced in colour, while their significance is explained and the motto, from Ovid, *Omnia Sunt Hominum Tenui Penitentia Filo* is translated as 'Man's All Depends Upon a Slender Thread'.

Founded in 1910 the Institute at first grew rather slowly. By 1925, when it received its Royal charter, membership was slightly more than 1,000. Following the war, however, there was a big increase, and there are now 5,639 members.

Expansion of the Institute's general activities was on a corresponding scale and administration was carried on under increasingly difficult conditions. It was not easy to find accommodation, and the premises

which now house the headquarters were only about 12 months ago used as warehouses and storerooms.

Transformation in that comparatively short time has been remarkable and the building now incorporates a lecture theatre with a seating capacity for 165, members' room, and committee room all on one floor, and also a library, dining-room and administrative offices.

A tablet to commemorate the work of the founders and their supporters was unveiled by the president in the entrance hall.

Rise in membership has been accompanied by a big increase in the Institute's educational work. Last year, there were 351 candidates (compared with the highest pre-war total of 241) for National Certificates in Textile's and the Institute examined 230 ATI candidates (more than in the last five pre-war years taken together) in general textile technology.

Work of the various committees has also grown and their meetings with those of the council are nearly 200 a year, twice as many as in 1946. The work includes granting of diplomas; publication of the Institute's *Journal*, text books and other textile works; unification of testing methods, textile terms and definitions and similar technical matters; award of scholarships and the adjudication of entries for the Institute's annual design competitions; and so forth.



# The World Shortage of Sulphur

## Economic & Scientific Factors Surveyed

**S**ULPHUR is one of the most important raw materials for industry, being used for the manufacture of sulphuric acid and directly or indirectly in the rayon, rubber, dyestuffs, wood pulp and many other important industries including agriculture. It has been said that the prosperity of a country might be gauged by its consumption of sulphuric acid. This is perhaps less true today, but nevertheless, it remains a fact that sulphuric acid is still used to a remarkable extent in most modern industries.

Many articles and technical papers have been published recently dealing from one aspect or another with the shortage of sulphur. It was thought useful to summarise the position in one paper and to indicate the lines on which work has been or is being done.

This has now been published for the Department of Scientific and Industrial Research (Intelligence Division) under the title 'A Survey of the Sulphur and Sulphuric Acid Position' (HMSO, 1s. 6d.; U.S.A., 40 cents), from information collated by R. Ashton, M.A., B.Sc., A.R.I.C., A. L. Thorogood, M.B.E., B.Sc., and D. Neville-Jones, M.A., B.Sc.

### America the Chief Source

Chief source of sulphur is the U.S.A., where it is obtained from the domes in Texas and Louisiana. Recent estimates of these reserves, however, suggested that there was only sufficient supply for 15 years at the present rate of mining. Restriction of internal consumption of sulphur and exports was therefore brought into force by the U.S. Government.

Exports of sulphur in 1951 were approximately 900,000 tons, of which the United Kingdom received some 400,000 tons.

In the first quarter of 1951, the original allocation of 81,465 tons to the United Kingdom was subsequently increased by 19,000 tons and an allocation of 95,000 tons for the second quarter led the Board of Trade to put into effect their proposed rationing scheme for an assumed consumption of 100,000 tons per quarter. In general, users received supplies varying from 80 to 90 per cent of their 1950 con-

sumption, except for the superphosphates industry which continued at two-thirds of its capacity and key industries—iron and steel, mineral and vegetable oil refining, explosives, etc.—which were maintained at full output.

For the third quarter the allocation was 106,300 tons, being 1,300 tons from indigenous production and 105,000 tons from imports. Of the quantity to be imported, 2,500 tons were to be provided by either Italy or Norway, but both countries were unable to supply it. For the fourth quarter, the allocation for import was 101,000 tons, of which 1,000 tons were to be provided by Italy, Norway or Mexico, the United States providing 100,000 tons.

### Three-quarters Used for Acid

Three-quarters of the imports were used for sulphuric acid manufacture and the remainder as a raw material for other chemical processes.

Pyrites form an additional important source of sulphur, the largest reserves being in Spain and Portugal. Large deposits also exist in the U.S.A. and Canada but neither have been developed on any scale.

Coal brasses—the pyrites content of coal—is another potentially important reserve, but the small content results in high production costs.

Of the eight British plants for the recovery of coal brasses which were in operation during the war, only three are now working—all in Lancashire. These produce some 6,000 tons per annum, containing 4.5 per cent carbon. It might be possible to increase this production to 8,000 tons per annum. Some 5,000 tons are also recovered by hand picking and this might be increased to 10,000 tons per annum. The material contains 12-14 per cent carbon and is not very suitable for roasting.

In the U.S.A., it is estimated that the amount of sulphur recoverable as coal brasses from washing coal in known deposits amounts to 3,000,000,000 tons, but the present recovery is negligible.

The only important raw material for sulphuric acid in Great Britain is anhydrite.

Extensive deposits occur at Billingham and in Cumberland and Furness.

A plant for production of sulphuric acid from anhydrite has been in operation for some time at Billingham. The present capacity of 100,000 tons of acid per annum is being extended by 73,500 tons. A new company, formed by 11 British firms, has a project to produce 150,000 tons per annum of sulphuric acid from anhydrite, but it will be some years before the plant can come into operation.

Other sources of sulphur discussed in the survey include: sulphate-reducing bacteria under examination at the Chemical Research Laboratory, DSIR; oil; oil shale and bituminous sands; natural gas.

Claims have been made over a number of years in respect of catalytic processes for the removal of sulphur compounds from coal and other gases. Some of these have been operated and others are being examined by the Fuel Research Station.

#### 'Ferralyt' Process

A new catalytic method—the 'Ferralyt Process,' details of which were first published in *THE CHEMICAL AGE*—is the subject of negotiation with various firms for its commercial application in the United Kingdom. At the time of the publication of the survey it was stated that pilot plants to assess the value of the process would shortly be in operation.

The 'Ferralyt' process may also be applied to flue gases, but the sulphur dioxide must first be reduced to hydrogen sulphide. A process has been developed by which the gases are passed with steam through a bed of hot coke forming hydrogen sulphide; which then goes through the normal 'Ferralyt' process.

However, in view of the extra materials and processing required it is considered probable that this scheme would be uneconomic compared with the recovery by washing with ammoniacal liquors, which also provides an outlet for the ammoniacal liquors from gas works and coke ovens.

In conclusion, the survey states that in the present year world production is 1,000,000 tons short of requirements and by 1953 the shortage, because of increasing demand, is expected to be about twice as great; this might be reduced to about 1,500,000 tons if possible gains from new reserves were allowed for. Thus, even with

the new supplies, it would seem that the world position, at least in the immediate future, will worsen.

The United Kingdom should therefore not relax its efforts to recover as much sulphur as possible from indigenous sources—anhydrite and coal—and to widen the basis of its supplies of sulphur and sulphuric acid from pyrites and oil. The possibility of doing this economically would, of course, depend to a large extent on the policy which the United States adopted on the price of sulphur.

Considering the conservation of sulphuric acid, it is apparent that, in the field of fertilisers, superphosphate will still be required. However, the use of alternatives, notably ground mineral phosphate, should be encouraged where suitable, and the manufacture of a nitrophosphate supplying both nitrogen and phosphorus should be started.

The use of ammonia from gas works and coke oven plants, either directly as a fertiliser or to wash the flue gases from power stations, could both economise in sulphuric acid and, at the same time, act as an amenity measure by reducing the amount of sulphur dioxide in the atmosphere.

#### $H_2SO_4$ & Uranium in S.A.

PLANS for the production of sulphuric acid and uranium are officially confirmed in the 1951 annual reports of the Daggafontein Mines, Ltd., and the Western Reefs Exploration and Development Company, Limited, of South Africa. Acid plants are stated to be under construction.

Plant to recover gold from the residues of the roasted pyrites accruing from the acid plant is also to be erected at a cost of £50,000 for each company. It is anticipated that the additional gold recovered will result in a satisfactory profit.

Under a revised agreement entered into with the Atomic Energy Board of South Africa, production of uranium, it is stated by both companies, will bring in a net profit somewhat greater than previously estimated.

Larger plants than originally projected will be constructed at both mines, and will, it is hoped, be in operation by 1953.

Shareholders will not be called upon to provide any of the finance required, while under the agreements the companies are relieved from any financial risks involved in the uranium production.



# The 'Autofining' Process

## A New Catalytic Desulphurisation Process

A NEW catalytic desulphurisation process, named 'Autofining' has been developed by the Anglo-Iranian Oil Company, it is reported. This process has been fully proved in extended operations in a 350-barrel-per-day pilot plant, and the first commercial-size unit of 3,500 barrels per stream-day input capacity, now being erected in the Anglo-Iranian Llandarcy refinery in South Wales, will be in operation shortly.

The 'Autofining' process operates at pressures of 50-200 lb. gauge and at temperatures of 700-800°F., and makes use of a very stable sulphur-resistant catalyst which has a long life. The catalyst is regenerated periodically, the on-stream period varying with the boiling range of the feed stock. A small amount of gas, consisting mainly of hydrogen, is produced in the process and this is recycled. No hydrogen manufacturing facilities are required.

A wide variety of feed stocks can be effectively desulphurised, ranging from natural and straight-run gasolines through naphthas and kerosines to gas oils and light diesel fuels. Sulphur removed from the feed stocks is largely converted into hydrogen sulphide. The products are of good colour and odour and are very stable. They need no subsequent refining treatment. The yields of liquid products obtained are 99 per cent plus

by weight or about 100 per cent by volume.

Straight-run gasoline and naphtha feed stocks having ASTM (American Society for Testing Materials) end-points of up to about 400°F. can be almost completely desulphurised by this process, and, at the same time, the octane number is raised by 2-3 numbers and the lead response considerably improved. With these feed-stocks catalyst on-stream periods of 800-1,000 hours between regenerations can be employed. Typical results on Middle East straight-run gasoline and naphtha are given in Table 1.

Kerosenes are also almost completely desulphurised by the 'Autofining' process and catalyst on-stream periods of about 400 hours between regenerations are easily achieved. Of particular value for certain markets is the improvement in burning properties (i.e., char value) obtained. Pilot plant results on a Middle East kerosene are given in Table 2.

Straight-run light gas oils (i.e., high-speed diesel fuels) may also be desulphurised. The degree of sulphur removal is lower than with lighter distillates but in the case of Middle East crudes, gas oils of 0.75 to 1.0 per cent sulphur content can be desulphurised to the extent of 60-70 per cent with catalyst on-stream periods of 200 hours. Typical results are reproduced in Table 3.

In some refinery situations it may be more desirable to desulphurise a long distillate and

AUTOFINING OF MIDDLE EAST STRAIGHT-RUN PETROL AND NAPHTHA

	Straight-Run Petrol		Naphtha	
	Feed	Product	Feed	Product
Yield per cent wt. on feed	99.5		99.7	
Sulphur removal, per cent.	99.1		98.4	
Catalyst on-stream period, hours	800		400	
Specific gravity, 60° F.	0.6957		0.7800	
Gravity ° API.	71.9	70.8	49.9	50.2
<b>Distillation:</b>				
I.B.P. ° F.	81	78	298	284
5 per cent. vol. at ° F.	104	102	308	303
10 " " " " " "	116	112	312	307
50 " " " " " "	200	200	332	329
90 " " " " " "	269	270	369	369
End Point ° F.	299	300	403	408
Sulphur per cent.	0.081	0.0007	0.116	0.0019
Octane Number (Motor)	60.0	62.3	—	—
" " + 1.5 ml TEL/IG.	69.0	75.0	—	—
" " + 3.5 ml TEL/IG.	75.0	81.0	—	—
Colour, Saybolt	ca. 20	30+	ca. 20	+ 26
Doctor Test	Positive	Negative	Positive	Negative
Corrosion Test	Positive	Negative	Positive	Negative

TABLE 1

## AUTOFINING OF MIDDLE EAST KEROSENE

Yield of product, per cent wt. . . . .	99.5	
Sulphur removal, per cent . . . . .	98.0	
Catalyst on-stream period, hours . . . . .	400	
Specific Gravity, 60° F. . . . .	Feed 0.7985	Product 0.7981
Gravity ° API . . . . .	45.7	45.8
<i>Distillation :</i>		
I.B.P. °F. . . . .	336	329
2 per cent vol. at °F. . . . .	352	345
10 " " " " . . . . .	365	363
50 " " " " . . . . .	404	404
90 " " " " . . . . .	446	448
End Point °F. " " . . . . .	471	476
Sulphur, per cent. . . . .	0.180	0.004
Mercaptan sulphur, per cent . . . . .	0.016	<0.001
Colour, Saybolt . . . . .	+15	+17
Flash point °F. . . . .	126	106
Odour . . . . .	Poor	Good
Doctor test . . . . .	Positive	Negative
Smoke point, mm. . . . .	25	26
Burning test (Institute of Petroleum) >		
Consumption g./hour . . . . .	20.6	20.1
Char value mg./Kg. . . . .	28	14
Appearance of glass . . . . .	Grey-brown film	Very faint grey film

TABLE 2

## AUTOFINING OF MIDDLE EAST VIRGIN GAS OILS

Yield of product, per cent wt. . . . .	99.1	
Sulphur removal, per cent . . . . .	70.0	
Catalyst on-stream period, hours . . . . .	210	
Specific gravity, 60° F. . . . .	Feed 0.854	Product 0.849
Gravity ° API . . . . .	34.2	35.2
<i>Distillation :</i>		
I.B.P. °F. . . . .	459	248
10 per cent vol. at °F. . . . .	520	505
50 " " " " . . . . .	561	556
90 " " " " . . . . .	619	612
End Point, °F. " " . . . . .	662	662
Sulphur, per cent . . . . .	1.00	0.30
Diesel index . . . . .	55	55
Aniline Point °F. . . . .	160	160
Carbon residue (10 per cent bottoms), per cent . . . . .	0.11	0.11

The above inspection data refer to the total liquid product, which contains a very small quantity of light ends. Removal of these light ends is easily accomplished in a simple flashing operation to give a product meeting specification flash point requirements.

TABLE 3

fractionate after processing, rather than desulphurise separate products after fractionation. Results obtained when processing long distillates from Middle East and Slaughter (West Texas) crudes in this manner are given in Table 4.

Other materials which may be satisfactorily desulphurised by the 'Autofining' process include the highly aromatic extracts obtained by the solvent extraction of petrol and kerosine fractions. (See Table 5.) The process is not normally applicable to residual fuel oils containing asphalt.

**Utilities Requirements.**—The expected consumptions of utilities in the 3,500-b.u.s.d. commercial unit now being erected at Llandarcy are given below:—

150 lb. Steam 6,600 lb./hour (net);  
Cooling water 30,000 Imperial gal./hour;

Fuel 25,000,000 B.Th.U./hour;  
Electric power 10 kilowatts.

In this plant the recycle gas compressor is steam-driven and steam is generated in a waste heat boiler.

**Investment Costs.**—The 3,500-b.p.s.d. commercial plant which was commissioned in 1951 will have a total erected cost within battery limits of approximately £250,000. Of this total cost, materials, including common building materials, draughting, engineering, procurement and contractors' overhead and profit, will amount to £196,000.

**Licensing.**—This process is covered by patents or patent applications in all major countries and is available for licensing by Anglo-Iranian Oil Co., Ltd., Britannic House, Finsbury Circus, London, E.C.2, England.



## New Nickel Alloy

### Result of Twelve Years Research

**B**ITAIN'S lead in gas turbine design has imposed upon metallurgists the need to produce materials with increasingly good properties at high temperatures. From the early days of the gas turbine engine, the Nimonic series of nickel-chromium alloys have become established as the standard materials for the rotor blades of every British aircraft gas turbine in production. To increase efficiency designers have been continually pressing for improved alloys which would give the required strength at ever increasing temperatures.

### Severe Conditions

When rotors are turning at speeds in excess of 10,000 r.p.m. and at temperatures substantially exceeding 800° C., the requirements of materials for the rotor blades are severe in the extreme. But in addition these materials, though giving the required strength and resistance to creep at high temperatures, must be capable of hot-working and machining.

The Birmingham research laboratories of the Mond Nickel Co. which early in the war years produced Nimonic, the nickel-chromium alloy which turned the gas turbine from an expensive prototype to a production power unit, have now announced a new advance, Nimonic 95. This alloy, which can only now be mentioned for the first time, has been developed from Nimonic 90.

Nimonic 95 is the result of intensive research which has continued for more than 12 years to improve the high-temperature properties of the nickel-chromium alloys without sacrificing forgeability at the expense of high-temperature strength. Improved methods of manufacture and processing have been adopted so that, although this new material is stronger and stiffer than Nimonic 90, it can still be hot-worked and machined.

A similar type of alloy to Nimonic 90, the new material has an increased content of hardening elements. It provides the same level of strength but operates at temperatures some 50° C. higher. The new alloy is already in production in the works of Henry Wiggin & Co., Ltd., and will soon be available in quantity.

## Industrial Library

THE American Library, at 41 Grosvenor Square, London, W.1, has opened an industrial section, which includes American books, periodicals, government documents, and other literature in the fields of technology, engineering, management, trade and agriculture.

Books and other material can be borrowed by anyone in Great Britain and, where necessary, will be sent by post. In cases where a book or periodical is not available for loan, photostatic copies of special articles will be provided free of charge. The Library also answers requests for information received by telephone or letter.

The Library works in close co-operation with British technical libraries and specialised research agencies, and acts as liaison with appropriate agencies in the United States where information in its own collection is not sufficient. It has full access to existing special American indexes which give complete reference by subject of all articles or books published in English.

## Contact Insecticide Research

RANDOLPH Riemschneider's comprehensive research work on contact insecticides, reviewed in *THE CHEMICAL AGE*, 65, 485-8, is to be published in book form by Paul Parey, Berlin SW. 68, Lindenstrasse, 44-47, under the title 'Scientific Research on the HCH— and Diene Groups,' by Dr. R. Riemschneider of the Institute of Chemistry at the Free University, Berlin. Subscription price for the 144-page volume is Dm. 22 (37s. 6d.).

This publication will be welcome in view of the increased number of scientific works on contact insecticides with a halogenated hydrocarbon base published in recent years, and because of the difficulty of forming a clear view of the field of synthetic insecticides. The book will present material recorded up to 1951, and contains over 3,000 sub-titles with details of reports, indications of the main subject (chemistry, physics or biology) and of the group of insecticides dealt with. The Diene group comprises insecticides of a more recent date, such as M 410, 'Chlordane', 'Aldrin', 'Dieldrin' and others. The insecticides of the terpene group ('Toxaphene', etc.) as well as benzene hexachloride and related compounds will also be dealt with.

## A Short Course in Microchemistry

By J. T. STOCK, M.Sc., Ph.D., F.R.I.C. & M. A. FILL, F.R.I.C.

FOR several years past the development of microchemical apparatus and techniques has been included in the research programmes of Norwood Technical College. Owing to the pressure both of teaching duties and of the ever-changing needs of a rapidly expanding College, much of the work has of necessity been simple in nature. It was therefore with considerable surprise that we received the invitation which led to the inclusion of a selection of original apparatus at a special exhibition held in the Science Museum, London, in 1950 (J. T. Stock and M. A. Fill, *Chemist and Druggist*, 1951, 155, 743). Partly as a result of the interest aroused by this exhibition, it was decided to arrange in this College an experimental short course of lectures and practical work illustrating various aspects of microchemistry. The course was successfully held on Saturday mornings in the Spring term of last year and is being repeated this year.

Since microchemistry is a big subject, the aims and objects of a course of total duration of but 36 hours naturally had to be limited. The policy adopted was to make the course essentially a practical one, to survey the principal branches of chemistry in which small-scale methods have been successfully applied and, with certain unavoidable exceptions, to concentrate largely on apparatus either easily constructed or normally at hand. Since the first group of students enrolled included representatives from such diverse fields as foodstuffs, teaching, pharmacy, metallurgy, and explosives, much useful discussion enlivened the lecture periods. The arranging of a limited number of basic experiments and a wide choice of optional ones certainly proved useful in catering for the divergent aims and interests of the students.

### Organic Applications First

**Small-Scale Organic Operations.**—Lectures and demonstrations were given by various members of the Chemistry Section of the College. Having surveyed the general aspects of microchemistry, some of the applications in the field of organic chemistry were examined first. The numerous standard operations, such as distillation, crys-

tallisation, solvent extraction and hydrolysis, involved in this branch of chemistry afford excellent illustrations of the important practical points involved in scaling-down the quantities of materials to be handled. The loss during working-up of a mere 20 milligrams of a product, so easily brought about by faulty technique, is obviously a serious matter when the maximum yield is only about a decigram!

### Several Advantages

Quite apart from those cases in which the adoption of micro-methods is rendered essential by the scarcity of sample, such methods have in certain cases the advantages of economy, speed and compactness over conventional procedure. It is in simple organic operations that the compactness of apparatus, both assembled and in store, becomes particularly apparent. Although it is not suggested that the complete elimination of macro-operations is either practicable or desirable, there is a good deal to be gained by the early introduction of semi-micro methods into courses in organic chemistry (R. Belcher and C. L. Wilson, *School Science Review*, 1948, 29, 161).

For some years past, we have made a practice of issuing to students standard sets of small-scale organic apparatus. Although of heavy Pyrex glass, such apparatus is no more expensive than cheap-grade macro-apparatus and, being much more robust, requires fewer replacements. Reaction flasks are of 20 ml. capacity, other apparatus being of corresponding scale. Typical items are the well-known filtering devices shown in Fig. 1. All are designed to operate under gentle suction and employ paper discs cut to appropriate size by means of a corkborer. The Willstatter 'nail' filter shown at (a) is of great general use, the King filter (b) being invaluable for 'inverted filtration', i.e., when a liquid is to be removed, leaving the bulk of the solid in the original vessel. Filtration through the Schwinger filter (c) is particularly useful for the isolation of a small quantity of a solid, since the latter collects as a compact column in the narrow throat.

Each set of apparatus is stored and issued

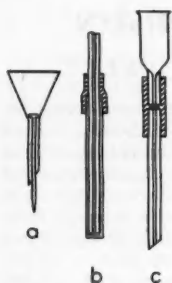


Fig. 1. — Filtering devices: (a) Willstatter 'nail' filter; (b) King filter; (c) Schwinger filter

in a separate numbered box of four compartments roughly 30 cm. square. Sides and partitions are of  $1\frac{1}{2}$  in. by  $\frac{1}{2}$  in. planed hardwood strip, the bottom being of 1/16-in. thick opaque Perspex sheet. Since the weight to be carried is very small, butt joints made with glue and nails are adequate. Construction is thus cheap, rapid and simple. The easy stacking of these boxes greatly facilitates storage of apparatus.

**Introductory Experiments.**—As introductory experiments, the conversion of one drop of aniline into pure acetanilide (J. T. Stock and M. A. Fill, *School Science Review*—in the Press) and the hydrolysis of 0.5 ml. of ethyl benzoate, followed by identification of the products, were carried out. For those wishing to concentrate on this field of work, a range of other experiments was available. Included were various reactions under pressure; these are often simple to perform on the small scale. As a substitute for the aluminium block normally used for heating the reaction tubes, we started by using a tin can packed with small nails. From this improvisation was developed the thermostatically controlled heating block (J. T. Stock and M. A. Fill, *Analyst*, 1951, 76, 497) shown in Fig. 2. Old cork borers, the lower ends of which are plugged with fine steel wool, form pockets for the reaction tubes and for the thermometer. For ease and safety in lifting in and out, reaction tubes are placed in similarly-plugged corkborers sliding easily into the pockets and having wire lifting loops. Temperature regulation is effected by expansion or contraction of air within bulb A, thus controlling the gas flow through the internally by-passed mercury cut-off. The temperature setting is adjusted by manipulation of screw clip B, which controls the normal height of mercury in the cut-off.

The conventional method of melting-point determination is, of course, a microchemical operation. Although fundamentally so simple, numerous points warrant attention. Examples are emergent thermometer stem correction, the handling of substances either thermally unstable or liquefiable only at high temperatures, and the procedure to be adopted when sharp melting at a definite temperature does not occur. Good examples of the latter type of substance are the fats and waxes or the camphor 'solutions' involved in molecular weight determination by the familiar Rast method.

A simple device for following melting or softening under controlled rates of heating is a melting-point tube of normal form,

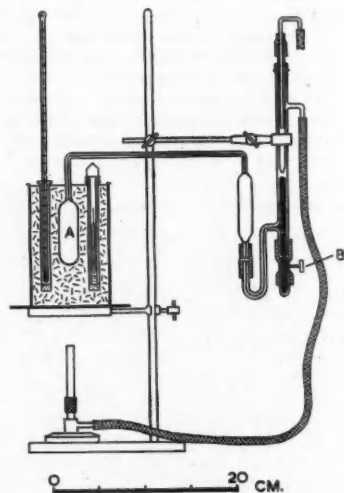


Fig. 2. — Thermostatically controlled heating block

except with the closed bottom made of blue glass. Having introduced the sample, a very fine glass rod having a ball-end of blue glass is inserted. When partial melting occurs, the rod sinks, finally reaching the bottom of the melting-point tube. The purpose of the coloured glass is to render the final stage easily visible. Fig. 3 shows an indicator in which the progress of softening or melting is signalled electrically (J. T. Stock and M. A.

Fig.

Fill,  
point  
mon  
glass  
down  
and  
upper  
twist  
enter  
long  
into  
lique  
seco  
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stage  
heig  
This  
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The  
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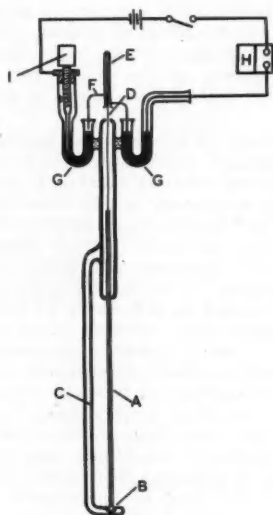


Fig. 3.—Alarm-type melting-point indicator

Fill, unpublished experiments). Melting-point tube A is supported alongside a thermometer in a depression B in the foot of glass frame C. Fine glass rod D, passing down the machine-drawn melting-point tube and resting upon the sample, carries on its upper end a light cap E. Round the cap is twisted contact wire F, the legs of which enter mercury cups GG. One leg is made longer than the other and dips permanently into the mercury. As rod D sinks into the liquefying sample the shorter leg touches the second mercury surface, completing the circuit and causing buzzer H to sound. The stage at which this occurs depends upon the height of the mercury in the left-hand cup. This height is governed by the position of screw I.

The separation of a mixture of volatile liquids into its components by fractional distillation is an important operation. On the micro and semi-micro scales, apparatus of quite high efficiency is often of fairly simple design. Nevertheless, many 'first attempts' in the practical periods demonstrated only too clearly that the efficiency of fractionation depends quite as much on proper operation as on proper design of equipment. In the same way, it soon became obvious that,

although ultimate combustion microanalysis is a rapid and accurate process in the hands of the practised, the acquisition of good technique does take a little time. However, the speed and ease of modern methods was generally appreciated even by those whose 'first time' figures appeared to disprove the generally accepted formula for sucrose.

**Inorganic Analysis.**—The complete change-over from macro to semi-micro qualitative inorganic analysis was made in this College in 1948. This step has never been regretted. The chemical aspect of the scheme used is the classical one, very sparing use being made of selective organic precipitants and other special reagents. The apparatus employed is very simple; a compact individual unit incorporating nearly all the necessary equipment has recently been developed (J. T. Stock and M. A. Fill, in course of publication).

On the small scale, the high area-to-volume ratio of the solution enables the latter to be rapidly and satisfactorily treated with hydrogen sulphide by mere agitation in an atmosphere of this gas (J. T. Stock and P. Heath, *Metallurgia*, 1950, 42, 44). Where the more usual method of saturation by bubbling

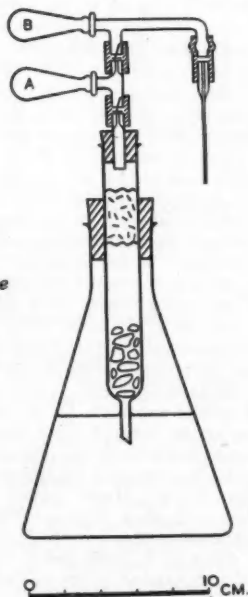


Fig. 4.—High-pressure hydrogen sulphide generator



is preferred, the small-bore delivery tubes employed require a fairly high pressure of gas (A. G. Lidstone, C. L. Wilson and D. W. Wilson, *Metallurgia*, 1947, **35**, 171; J. T. Stock and P. Heath, *Ibid.*, 1950, **41**, 171; J. T. Stock, P. Heath and W. A. L. Marshment, *Ibid.*, 1950, **41**, 345). A recently developed generator for this purpose is shown in Fig. 4 (J. T. Stock and P. Heath, *Analyst*, 1951, **76**, 496). Generation of gas does not occur until the gentle squeezing and releasing of teat A has drawn up the acid into the iron sulphide container. Further operation of A expels air and delivers hydrogen sulphide under pressure high enough to permit a delivery tube having an orifice of only about 0.1 mm. Under these conditions the upper teat B has sufficient reservoir effect to allow continuous bubbling to be maintained by compressing A only about twice per minute.

#### Students Given a Trial

Following a demonstration of the complete identification and confirmation of the components of a typical 'four-radical mixture' or of a typical non-ferrous alloy, similar substances were made available to students wishing to try for themselves. As an illustration of the useful technique of working on filter paper strips, the separation of copper, cadmium and bismuth was used.

Various types of micrometer and other microburettes were demonstrated in a general survey of inorganic quantitative work. Possibilities of working in highly dilute solution were considered as an alternative to the handling of minute volumes of more concentrated solution. As a practical exercise, the indirect micro-determination of barium by iodometry served to stress the advantage of a process having a favourable equivalent. This was followed by a gravimetric determination of the same metal, illustrating the technique of accurate weighing, and the need for correct precipitation conditions.

**Physico-chemical Technique.**—Various forms of apparatus for the determination of molecular weights were demonstrated and made available in the practical periods. For volatile substances an apparatus working on the Victor Meyer principle (A. Adair, *School Science Review*, 1949, **30**, 240) has undergone considerable development in the College (J. T. Stock and C. Heitler, in course of publication). The Dumas method although involving greater manipulative (and

arithmetical!) ability, requires the simplest of apparatus. A soft glass ampoule makes a useful micro Dumas bulb, being light and sitting squarely on the balance pan (J. T. Stock and P. Heath, in course of publication).

Such methods are, of course, inapplicable to substances which cannot be volatilised. In such cases, methods based on the properties of solutions come into their own. Several forms of ebulliometric apparatus were in use to illustrate one aspect of these properties. Included was the compact Cottrell-type unit shown in Fig. 5. In this, the pump assembly is arranged to slide easily on the stem of the thermometer, ensuring accurate alignment. Escape of vapour is prevented by a cold-finger condenser supported in the side tube down which samples are introduced (J. T. Stock, *School Science Review*—in the Press). In highly dilute solutions, the very small elevation of the boiling point may, in principle, be measured electrically by replacing the thermometer by a thermistor. The necessary conditions for obtaining satisfactory degrees of accuracy and reproducibility have received considerable attention. Using the amplifier-recorder unit of the College's Tinsley recording polarograph, it has proved

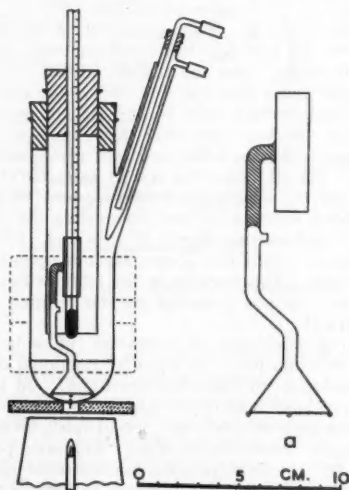
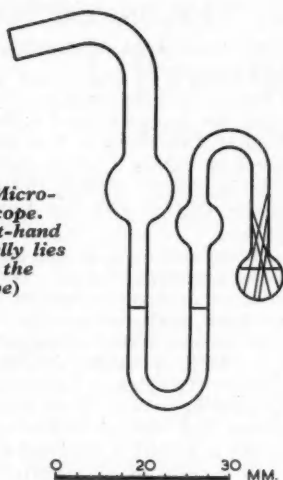


Fig. 5.—Cottrell-type molecular weight apparatus—pump tube shown enlarged at (a)



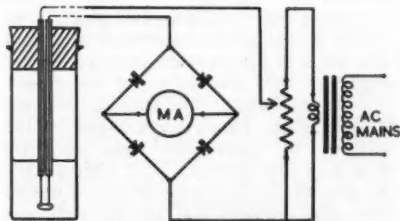
**Fig. 6.—Micro-isoteniscope.**  
(The right-hand bulb actually lies behind the V-tube)



possible to record the progress of certain chemical reactions occurring in boiling solution. (Unpublished experiments).

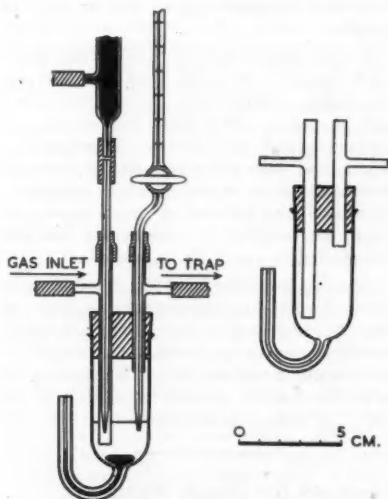
The measurement of vapour pressure over a range of temperatures is a matter of considerable importance, particularly in connection with the evaluation of latent heats of vaporisation. Used in conjunction with ancillary measuring apparatus, the easily constructed Steinbach-Devor micro-isoteniscope (O. F. Steinbach and A. W. Devor, *J. Chem. Ed.*, 1945, 22, 496) permits the plotting of a vapour pressure curve with less than 1 ml. of liquid. The fine glass rods shown in Fig. 6 are inserted before bending-up and are intended to promote smooth boiling.

A simple direct-reading conductometric



**Fig. 7.—Conductometric apparatus for the study of reaction kinetics**

micro-titration apparatus (J. T. Stock, *Metalurgia*, 1950, 42, 48), first exhibited at a meeting of the Micro-chemistry Group of the Society of Public Analysts and Other Analytical Chemists, enabled students to explore the possibilities of this particular field. A slightly simplified version, shown diagrammatically in Fig. 7, enabled another use of conductometry, viz., the study of the kinetics of hydrolysis of such compounds as tertiary butyl iodide, to be tried out (J. T. Stock, *School Science Review*, 1950, 31, 336). The production of highly conducting hydrogen iodide during the reaction enables the progress of the latter to be followed by mere observation of the milliammeter. The elec-



**Fig. 8.—Cell for polarography and amperometric titration**

trode system consists merely of a pair of melting-point tubes carrying short pieces of platinum wire which are platinised before use in the usual way.

The introductory aspects of certain other branches of electrochemistry, such as potentiometry and polarisation, are being included in the current course. Although not specifically designed as a microchemical tool, a home-made direct-reading pH meter is useful in illustrating the principles involved in potentiometric titration (J. T. Stock, *School Science Review*, 1951, 32, 287).

Polarography and related techniques are now so important and are so essentially microchemical in nature that their inclusion, however limited, in the course seemed desirable. The construction of polarograms of oxygen dissolved in potassium chloride solution, and the amperometric titration of milligram quantities of nickel by dimethylglyoxime solution were used as examples. The simple cell shown in Fig. 8 was used. The outer portion is made from a 1 in.-diameter Pyrex boiling tube (M. A. Fill and J. T. Stock, *Analyst*, 1944, **69**, 178). When the dropping mercury electrode and burette jet are thrust down the tee-pieces, the clearance is ample for the passage of the gas stream used for deoxygenation or for stirring the solution.

**Microscopy.**—The microscope has many uses, including the determination of refractive index, identification of certain organic and inorganic substances, examination of mixtures, and particle-size determination. The last of these was illustrated by examination of a sample of poorly refined chocolate. In view of the presence of large crystals of sugar the roughness to the palate was not unexpected.

The course is scheduled for repetition next year. If the interest continues to grow, it may be necessary to bring into use extra laboratory accommodation, or to duplicate the present arrangement. The running of extension courses, already the subject of inquiry, is under consideration.

## Inquiry for Back Numbers

We have had an inquiry from a public library in South Africa which requires the following copies of *THE CHEMICAL AGE* to complete its files for binding:—1921. 22 January, 12 February, 12 March, 30 April; 1922: 26 August; 1923: 6 January, 17 February; 1924: 12 July, 2 August; 1925: 3 January, 30 May, 8 August, 24 October; 1940: 6 January; 1941: 26 January; 1942: 7 February; 1944: 2 December. Indexes to Vols. 4, 14, 40, 43, 44 and 50.

If any of our readers have any of these copies and do not require them, we should be grateful. The library concerned is willing to pay for them if necessary.

## Transparent Metal

### NPL Develops Method of Coating Glass

A METHOD of coating glass with a thin, transparent film which will conduct electricity has been developed at the National Physical Laboratory. The film can be heated by passing a current through it. It could be used, for example, in aircraft wind-screens, to keep them free from ice and snow.

The layer on the glass is an extremely thin one of an oxide of a metal. Not all metals produce transparent oxide films. One which has given good results is tin. It can be deposited very evenly on the glass by techniques which are already well understood in industry. The glass is then heated to near the softening point and cooled again. The tin oxidises and becomes transparent. Finally the film is washed in water and dried, a step which increases its conductivity. After this it is hard and inseparable from the glass surface and it is resistant to chemical attack.

### Visibility Little Reduced

Visibility through the glass is reduced by the metal coating by an amount which is negligible for all practical purposes. The coating has a resistance of about 1,000 ohms between the opposite edges of any square area. Enough current can be passed through it to keep the surface of the glass so hot that it is impossible for ice and snow to form on it or for condensation to make it misty. In practice the film would be sandwiched between two layers of glass.

The treated-glass may also be suitable for car windcreens and for shop windows to keep them from steaming over in cold weather.

Ceramic materials as well as glass can be treated by the process, and there are many uses for it in the electrical industry, for instance in the production of fixed and variable resistances. The glass can be used to avoid static charges accumulating on the windows of instruments when they are cleaned in dry weather. This static electricity can lead to false readings and it was to solve this problem that the research was originally begun at the NPL.

The process is being patented.

## MIDLANDS SOCIETY FOR ANALYTICAL CHEMISTRY

# Ultramicroanalysis

## Past, Present and Future—I

AT a recent meeting of the Midlands Society for Analytical Chemistry held in Birmingham, Dr. C. L. Wilson, Reader in Inorganic and Analytical Chemistry, Queen's University, Belfast delivered a lecture entitled, 'Ultramicroanalysis, Past, Present and Future.'

Dr. Wilson began by stating that ultramicroanalysis takes its place as the newest branch of analytical chemistry. As the name indicates (he said) it deals with the analysis, qualitative and quantitative, of microgram amounts of material. Entirely new techniques and apparatus have been devised, but these are by no means outside the scope of laboratories which normally cover the different fields of microanalysis. Ultramicroanalysis has provided, and will continue to provide, the analytical chemist with a most powerful tool.

After outlining the objects of his address, Dr. Wilson continued:—

The approximate sample sizes required for a particular scale of analysis are summarised below, as well as the comparative volumes used in titrimetric analysis on similar scales.

### Sample Sizes

	Sample size (g).	Titration Volume (ml.)
Macroanalysis	$10-(5 \times 10^{-2})$	250—10
Semimicroanalysis	$(5 \times 10^{-2})-(5 \times 10^{-3})$	10—0.5
Microanalysis	$(5 \times 10^{-3})-10^{-4}$	0.5— $(2 \times 10^{-2})$
Ultramicroanalysis or Microgram Analysis	$10^{-4}-10^{-7}$	$(2 \times 10^{-3})-10^{-4}$

The quantities involved in ultramicroanalysis are thus exceedingly small. However, more than sufficient molecules are present to ensure that the limit of identification is not exceeded in a qualitative analysis, and in many cases to permit an accurate quantitative analysis to be made. Indeed, Benedetti-Pichler, one of the pioneer workers in the field of ultramicroanalysis, has calculated that the limit of identification of a substance is, in general, about  $10^{-20}$  g., while a quantitative analysis could probably be effected on as little as  $10^{-16}$  g. with a reasonable degree of reproducibility.

With amounts less than these, we are

dealing with molecular quantities and the question of the chemical kinetics of the reaction involved becomes of the utmost importance. It must be remembered that analytical processes are, in the main, concerned with immediate reaction, and this, of course, is closely connected with chemical kinetics. Consider, for example, the precipitation of silver chloride—differing techniques, such as are encountered in organic preparative chemistry, may have to be applied to obtain quantitative precipitation of microgram and sub-microgram amounts. As far as the chemistry of the precipitation is concerned, however, if we can develop a suitable technique to cope with it, then the normal reactions will hold. This reasoning may be applied to other chemical reactions.

Analysis by spot tests is perhaps the earliest example of qualitative ultramicroanalysis.

### Emich the Pioneer

Emich is considered the pioneer worker and founder of microchemistry as a science, though qualitative identification by means of microscopic crystal tests had been placed on a firm footing by earlier workers such as Behrens and Streng. Emich, however, was the first to discuss the practical and theoretical limits of sensitivity. He was, indeed, the first to attempt general and physico-chemical experimentation on a small scale, and by 1911, when his 'Lehrbuch der Mikrochemie' was published, microchemistry was an established science. In collaboration with Donau, Emich developed qualitative ultramicroanalysis using a microscopic technique, and using a fibre technique he proposed certain confirmatory tests which had limits of identification of the order of  $10^{-10}$  g.

Wigglesworth (1927), working on 0.002 ml. of fluid, developed a technique for obtaining an accurate determination of the hydrogen ion concentration in the alimentary canal of the cockroach. The essential feature of his technique was the use of waxed plates and capillary pipettes with the consequent elimination of the capillary of

the fluids examined. The waxed capillary pipette was filled with fluid to an arbitrary graduation mark and a solution of an appropriate indicator run in until a second arbitrary graduation mark was reached. After mixing, the pH of the fluid was determined by comparing it with mixtures made up with the same indicator solution in the same pipette, using standard buffer solutions in place of the test fluid. The colour comparison could be effected by expelling the drops of fluid on to a white tile covered by a film of wax.

### Titrimetric Apparatus Developed

A further advance in the techniques of ultramicroanalysis was the development of titrimetric apparatus and procedures by Kirk and his co-workers. This work has been fully described in a recent text (1950); consequently only a brief account of it need be given here. There is no doubt that the most accurate quantitative aspect of ultramicrochemistry is titrimetric analysis, because very small volumes of liquids may be easily and accurately measured and handled. Thick-walled capillary pipettes are normally used for measuring and delivering liquids.

A useful pipette has been designed by Lisco, Cunningham and Kirk (1941) for use in biological research. The pipette is controlled by a syringe and consists simply of a capillary tube with two bulbs, one acting as a container and the other as a trap. A hypodermic syringe has been found to be most convenient as a control for the pipette. The pipette must, of course, be calibrated. This is done by filling it with mercury to the calibrated mark, and then expelling and weighing. The necessary corrections for temperature are made and the weight of mercury related to its true volume.

Burettes for delivering measured amounts of liquids are also conveniently constructed from capillary tubing, so that a small volume of liquid occupies a comparatively long portion of tube. Such capillary burettes contain up to 1 ml. of solution, and are best filled or emptied by using a moving thread of mercury controlled by a screw plunger device. Horizontal-type burettes are preferred for ultramicro titrations especially if they are to be used in conjunction with a microscope.

Kirk used special white porcelain dishes as titration vessels which have many advantages over beakers or flasks. Stirring may be achieved by means of a magnetic vibrat-

ing thread stirrer, or, on occasion, by means of a stream of gas.

Special titration vessel supports and illumination are necessary aids in ultramicro titrimetric work. The latter is of particular value, as it is generally extremely difficult to detect end-points in small volumes of solution.

A considerable literature exists on the qualitative and titrimetric analysis of microgram amounts of materials. Only comparatively recently, however, have satisfactory gravimetric procedures been reported, because of the lack of sufficiently reliable balances for such small-scale work. It is clear that balances having a sensitivity of at least  $\pm 10^{-8}$  g. and able to carry a load of 100 mg. or more are necessary for accurate quantitative gravimetric work. Most of the ultramicro gravimetric methods recommended so far are, indeed, semi-quantitative. So, for work of the highest accuracy a balance is desired which will weigh microgram samples with an accuracy comparable to that obtained with the analytical or microchemical balances on proportionally larger samples.

The quartz microgram balance developed by Kirk and his co-workers (1947) for work on the transuranic element plutonium fulfilled these requirements, and in addition it was claimed to be more practical for routine use than earlier types of balance. Principles incorporated in certain of the earlier balances were used; e.g., the pan suspension of the Steele-Grant balance (1909) and the pan well of Pettersson (1914). The balance was of the torsion type, and utilised the optical system of Neher (1942) to increase the relative sensitivity.

### First Micro Balance

The first micro balance, of the so-called displacement type, was described by Salvioni (1905). The principle of this balance was subsequently used by later workers in the construction of true microgram balances. Briefly the principle of Salvioni's balance was as follows: The beam was a glass thread carrying a pan at one end and fixed at the other. The displacement of the free end with different loads on the pan was measured using a travelling microscope to effect the reading. In general the highest sensitivity obtained with this type of balance was about  $10^{-6}$  g., and the load capacity varied from about 1 to 200 mg.

Riesenfeld and Möller (1915) and Emich

and Donau (1915) improved the classical torsion-displacement balance of Nerst (1903) and obtained a sensitivity of  $10^{-7}$  g. on a 15 mg. load.

A third earlier microgram balance was the beam-knife-edge type of which the Steele-Grant (1909) balance was perhaps the outstanding example. The balance was of classical design but had a beam consisting of quartz rods fused together attached to a quartz knife edge on a quartz plane. The movement of the beam was followed by means of a mirror-light ray device, the mirror being fused at one end of the balance beam. At the other end of the beam were a quartz bulb, a quartz pan and a hook for carrying counterpoises. The balance was placed in a case which could be evacuated. After loading the pan, the balance was restored to zero by altering the pressure within the case. This altered the buoyancy of the quartz bulb and the pressure change necessary could then be related to the pan load. This balance could carry a load of 20 mg. and had a sensitivity of  $4 \times 10^{-9}$  g. In the Ramsey-Whytlaw-Gray (1912) balance, of the same type, the load was increased to 50 mg. and the sensitivity decreased to  $2 \times 10^{-9}$  g.

The Pettersson balance had quartz fibre in place of knife-edge suspension to overcome dust effects. Again, the pans were placed in separate compartments in order to leave the beam housing intact.

A quartz spring or helix type microbalance was favoured by Emich (1915). It could be employed, among other things, for rapid weighing and was most convenient to use. Its main disadvantage was the difficulty in the drawing of the quartz fibres and the winding of the helix, while the sensitivity was somewhat less satisfactory than with other types of microgram balance.

Finally, the torsion-restoration balance of Kirk and his co-workers incorporated, as previously mentioned, some of the outstanding features of the Neher, Steele-Grant and Pettersson balances.

#### Benedetti-Pichler's Work

Ultramicro methods of analysis are extremely useful where it is desired to keep the sample intact. Thus, they have proved of immense value in the examination of old paintings. Benedetti-Pichler (1937) analysed the pigments used in the fossae of the incisions of Chinese oracle bones using ultra-

microchemical techniques, and claimed that such a small amount of material was used in the chemical analysis that even microscopic inspection of the specimens would be unable to prove that an analysis had been carried out.

In the same year, Benedetti-Pichler described a general working technique for the qualitative analysis of microgram solid samples. Emich and other workers had, of course, previously effected certain relatively simple analytical procedures on samples less than 1 microgram. The Benedetti-Pichler technique involved working in capillary cones of capacity 0.5 cu. mm. and observing by means of a low power microscope, most qualitative operations on the microgram scale requiring continuous microscopical observation.

#### Evaporation Prevented

The reagents and most of the apparatus used were assembled on a small glass slide, which was placed in a moist chamber which prevented the evaporation of the minute volumes of solutions used. For centrifuging, heating, etc., the capillary cones containing the solution were transferred to capillaries designed to minimise evaporation. The transfer of solutions was performed using a syringe capillary pipette technique. Benedetti-Pichler described working procedures for the qualitative analysis of 0.01 cu. mm. of a solution containing 0.1 microgram of antimony and 0.01 microgram of bismuth. He gave details of ultramicro techniques for the passage of  $H_2S$  gas, the semi-quantitative determination of the precipitate, the separation of solution and precipitate, and the heating and preserving of solutions in capillary cones.

More recently, Benedetti-Pichler and Cefola (1942) extended the technique of working in the capillary cone to permit the carrying out of confirmatory tests after the separation of constituents. It was found possible to identify approximately 0.001 microgram of the more common ions of the hydrogen sulphide group. Certain improvements were also made to the moist cell used in earlier work, with the result that a greater variety of tools could be accommodated.

A later paper by these workers (1943) described the application of capillary cone techniques to microgram analyses requiring lengthy separations followed by sedimentic determinations and confirmatory tests. In

particular, a clear-cut separation, estimation and identification of the more common ions of the hydrogen sulphide group was achieved. A technique was described whereby distillation could be effected from one capillary cone to another.

#### Atomic Energy Work

Extensive ultramicroanalytical work was carried out by atomic energy workers during the war and subsequent years. Indeed, but for ultramicroanalysis the chemistry of the transuranic elements could not have been elucidated and the atomic project could not have been achieved.

Although most of this work has not been published because of security reasons, nevertheless the comparatively small amount published reveals that some ingenious ultramicrotechniques have been devised to cope with the chemistry involved. Of particular value is the text edited by Seaborg, Katz and Manning (1949) which described some of these techniques in a series of papers on the chemistry of the transuranium elements; for example, special techniques are described for the electrodeposition of plutonium, and for determining the oxidation-reduction potential of this element.

*(To be continued)*

## Handling Electric Cable

### Value of Contactless Self-Winding Reels

OF interest to many branches of chemical engineering is the latest 'Wayne' range of patent contactless type self-winding electric cable reels.

These contactless designs are particularly valuable in the case of bad operating conditions, such as when large quantities of injurious dust may be present, or in cases where the atmosphere is vapour bound, due to process work or outdoor situations. In laboratory work where highly inflammable vapours are present or when handling explosives, the contactless design is, of course, highly desirable, if in fact not essential.

Still another application is electric lifting magnets in which the safety factor in providing current to the magnet must be kept very high in order to eliminate any risk of current failure while the magnet is being used, and probably swinging a load of steel over delicate machinery, or in other situations where a failure in the load would have serious results.

The reels, which are available in a number

of types and sizes, are produced by Power House Components, Ltd., Nottingham, and carry the registered trade mark 'PHC.'

General principle of the standard reel is similar to that of the spring roller blind, the cable being pulled out as required against the action of a spring which ensures that it is kept taut and when released winds smoothly back again. This results in a great improvement in the working of electric cables of all kinds, including reduced wear and tear and maintenance costs.

From this basic principle, however, many important developments have gradually been evolved, including particularly the 'Wayne' patent trailer (contactless) type. This is of the double type which pays out two lines of cable in opposite directions, so that the reel can be placed in the centre of the work and double the amount of cable handled because the pay-out is in two directions at once, while current-carrying slip-rings are eliminated.

The reel positions itself at points midway between the static electrical connection and the equipment being served, such as for example, an electric hoist, but it must move along the rolled steel joist which carries the hoist, or otherwise the cable attached to the supply point would sag as it is paid off the drum. An important feature is that the rubber cable is not broken between the point of contact with the machine being supplied with current, and at the other end of the cable where it is connected to the electric supply point.

### U.S. Steel in 1951

The U.S. steel industry spent more than \$1,000,000,000 on modernisation and expansion in 1951 and reached a record steel capacity of 110,000,000 tons per year, according to the March issue of *Steelways*, the magazine of the American Iron and Steel Institute. A greater amount will be expended in 1952, to bring the total expense for these items to \$5,000,000,000 since 1946; the goal is to bring capacity to 120,000,000 tons in 1953. *Steelways* points out that the industry is developing its sources of iron ore on an equally large scale. Rich ore beds in Venezuela, Canada and Liberia are being opened up, and their output will be supplemented by extensive U.S. low-grade deposits which can now be worked as a result of new processes.



# R.I.C. Annual Report for 1951

## Council Outlines Wide Scope of Activities

THIS year the annual meeting of the Royal Institute of Chemistry is being held in Dublin on 18 April and the annual report of the Council, to be presented then, has already been published. Amongst the many interesting facts revealed in this report is that membership of the Institute increased more rapidly in 1951 than in recent years, there now being 10,797 members in the United Kingdom and Ireland.

The year 1951, states the Council, began with the allowance by the Privy Council on 19 January of the new By-laws. Thus was completed a process involving the application for a new Royal Charter and its granting by H.M. King George VI in 1949 and the complete revision of the By-laws.

These matters had been among the major concerns of the Council and Local Section Committees for over five years and their completion was therefore a matter of great satisfaction.

One of the results was to empower the council to elect persons of distinction, not necessarily chemists or of British nationality, as Honorary Fellows of the institute, subject to a limit of 20 at any one time.

### First Honorary Fellow

The Duke of Edinburgh had accepted the invitation of the council to become the first Honorary Fellow. The interest His Royal Highness takes in scientific matters is well known and was exemplified by the success with which he discharged his duties as president of the British Association for the Advancement of Science. It was therefore felt most fitting that he should become associated with the professional organisation for one of the major branches of science.

Membership of the Institute, according to the annual report, had increased more rapidly than in recent years. The total number of members in Great Britain and Ireland was now 10,797, of which the largest sections were: London and South-Eastern Counties 4,270; Manchester and District 945; Liverpool and North Western 891; and Birmingham and Midlands 726.

Overseas Sections totalled 530 members of which 324 were in India, 96 in New Zealand,

78 in the Cape of Good Hope and 32 in Malaya.

Entries to the Associateship examination had continued to rise, but the proportion of successful candidates was lower than in the previous year.

### Reserves Built Up

As a result of economies and certain increases in fees the excess of income over ordinary expenditure had been raised sufficiently to enable more appropriate sums to be set aside as reserves for intermittent publications and general contingencies. With continually rising costs, however, it would clearly become difficult to maintain such provisions in future and the council was therefore giving serious consideration to means of balancing its budget in 1952.

During the year important developments had taken place in the organisation of Institute affairs in India. The new scheme for forwarding applications for admission by candidates who had received their training in India or Pakistan, had been brought successfully into operation. Serious attention had been given to the possibility of holding examinations for the Associateship in India, but the machinery for doing so was still under discussion at the end of the year.

### Joint Subscribers Unchanged

The number of joint subscribers to at least the three chartered chemical bodies under the Chemical Council scheme was practically unchanged from that in the previous year—3,558 against 3,546; Fellows and Associates accounted for 3,470 of the number for 1951. This represented nearly 45 per cent of the total membership of the Chemical Society and almost half of that of the Society of Chemical Industry—but less than a third of the corporate membership of the Institute.

It was regrettable that, in spite of the attractions of the joint subscription scheme, more than half the members of the Institute did not support either of the principal publishing societies or the Faraday Society or the Society of Public Analysts and Other Analytical Chemists. On the other hand, some satisfaction might be derived from the estimate that the roll of the Institute prob-

ably included over 70 per cent of the chemists in the United Kingdom who were qualified for admission to membership.

Need for chemists with experience, particularly in some special fields, had continued to grow, as was witnessed by the considerably larger number of vacancies notified. It was satisfying to find that there had been a marked increase in the number of senior vacancies brought to the notice of the Institute, especially by firms employing Fellows or Associates in administrative positions. In fact, the demand for most categories of qualified scientists, including chemists, continued to outstrip the supply, in spite of the great expansion in their number that had occurred since the war.

The importance of making the best use of available scientific personnel, especially in relation to requirements arising from the defence programme, had been recognised by the Government in reconstituting the Technical Personnel Committee (again under the chairmanship of Lord Hankey) which was in operation during and immediately after the war.

#### Salaries Increased

Salaries of chemists had increased notably since the last issue of Remuneration Statistics by the Institute and it appeared that the average figures then quoted for any particular age group might now apply more closely to the immediately lower age group. In order to be able to assess the position more exactly it had been agreed to conduct a further survey of salaries and to publish revised remuneration statistics as soon as possible.

Professor J. W. Cook, in his presidential address on 'Some Thoughts on the Education of the Chemists', delivered at the annual general meeting in April, drew attention to a number of aspects of education and training in chemistry and ancillary subjects. Subsequently the council decided to set up an Education Committee to review and report on matters of interest to the Institute in this field.

So far the committee had been largely concerned with questions of courses and examinations for National Certificates but latterly had been turning its attention to the teaching of chemistry in schools. It was believed this committee would afford a useful link with teachers in technical colleges and schools on problems that were of great importance in relation to the Institute's own qualifications.

Preparation of a new edition of *The Profession of Chemistry* was not completed during the year, and it was decided in the meantime to produce two pamphlets, 'How to Become a Chemist' and 'Careers in Chemistry'. These would be available in the office, primarily as an aid to answering inquiries from schoolchildren, parents and teachers.

In the early part of the year further consideration was given to the recommendations on the future of higher technological education made by the National Advisory Council on Education for Industry and Commerce. A memorandum on this subject was submitted by the council of the Institute to the Ministry of Education, and representatives of the Institute discussed with officers of the Ministry the criticisms of the recommendations that had been made in the memorandum. The final report of the National Advisory Council, issued later in the year, went some way towards meeting certain of these criticisms, but the Institute council was still not satisfied that a case had been made for going on with the project of establishing a College of Technologists as an awarding body.

It was noted with satisfaction that courses for the training of laboratory technicians had been established at the L.C.C. Paddington Technical College and were being developed at other centres, to prepare candidates for the examinations of the City and Guilds of London Institute in Laboratory Technicians' Work. The Royal Institute of Chemistry had been represented on the various committees that explored this field and eventually drew up syllabuses for the courses, and the council attached considerable importance to the further development of this project.

#### Increase Expected

Reports from Dusseldorf last week indicated that an early increase in the price of steel by approximately £5 a ton and the freeing of steel scrap from controls was confidently expected by German industrialists. Meanwhile the German steel industry set up a new post-war record in March with an output of 1,318,000 tons of crude steel compared with 1,027,000 tons in the same month of last year.



## Chemistry & Education

### Stranglehold of Administration

EDUCATION was the main theme of the speeches at the annual dinner of the London Section of the British Association of Chemists held on Friday, 28 March.

Proposing the toast of the Association, the chairman, Mr. H. T. F. Rhodes, Dip.I.C. (Lyon) said that the BAC had done much to gain greater appreciation of the chemist and proper recognition of his status. In the London Section it had always been the aim to try and get hold of the young people and encourage them to become members. He regretted that there were not more young people present.

A new effort to arouse interest in the BAC had been a successful meeting at which the secretaries of the technical colleges in London had been entertained and an arrangement was proposed whereby the secretaries could form a group membership. It was felt this might prove beneficial to members of the colleges as well as introducing new blood into the BAC.

Strong views on education and its importance not only to the future of chemistry but to science generally were expressed by Mr. H. L. Howard, B.Sc., A.R.C.S., D.I.C., M.I.Chem.E., F.R.I.C., hon. registrar of the BAC. Much had been written and spoken in recent years about higher technological education, but far too little attention had been given to the ordinary administrative aspect and to the lack of staff—it was estimated that by 1956 there would be about 5,000 too few in the teaching profession.

### Unnecessary Administration

Education was at present being strangled by 'red tape' and unnecessary administrative problems. The modern teacher could not devote adequate time to his subjects or students because of the hours which had to be spent as an 'office boy'. He himself after 25 years of part-time teaching felt that 95 per cent of his time was now wasted and he felt the moment had come when some definite action was necessary. He had therefore resigned his post as head of the science department of East Ham Technical College.

In conclusion, Mr. Howard urged that chemists should get together on this vital matter of education, and he hoped it would be possible to arrange some form of joint discussion in the not-too-distant future.

Replying to the toast of 'The Guests,' Dr. C. W. Herd, chairman of the London Section of the Royal Institute of Chemistry, said it was a pity more people did not have the courage to support their convictions with action like Mr. Howard. There were bound to be problems connected with the administrative side of education, but these must be overcome and not allowed to stifle the scientist in his work.

There was no doubt as to the usefulness of chemistry in life—it appeared everywhere. It was up to the chemists to set an example in the better appreciation of chemistry in industry, education and other services. Through the work of the BAC there was an increasing appreciation of chemistry's value to industry, and more chemists were being appointed to executive positions.

The medical profession tended to be held in more esteem by the public because it was more directly in contact with them. Yet how seldom were the advances in medicine due to chemists recognised?

More appreciation of the chemist should be encouraged and his status would then be improved.

### Ceramicists' Meeting

The spring meeting of the Refractory Materials Section of The British Ceramic Society will be held in the library of the Royal Sanitary Institute, 90 Buckingham Palace Road, London, S.W.1, commencing at 10 a.m. on Thursday, 5 June. The technical papers to be presented and discussed will be concerned with the 'Mechanism of Sintering', the 'After-Contraction Test Applied to Firebricks' and the 'Mineralogical Constitution of Fireclays'.

### Morphine Synthesised

The synthesis of the alkaloid morphine—age-old problem in organic chemistry—is reported to have been achieved by two chemists at the University of Rochester, U.S.A. The starting material was Schaeffer's Acid (2-naphthol-6-sulphonic acid), and the total synthesis involved 27 steps and proceeded *via* codeine. Dr. M. Gates, who, with Dr. G. Tschudi, was responsible for the synthesis, began work on the process four years ago. It was financially supported by Merck & Co., and the Research Corporation of New York.

## Education & Plastics

### Training Grants to be Awarded this Year

TRUSTEES have now been appointed for the Plastics Industry Education Fund. This came into being last September to continue the work of the 'President's Fund' established in 1942 by Mr. Kenneth Chance, of British Industrial Plastics, for the promotion of education within the industry.

Formally constituted on 6 March, 1952, the trustees, who were selected after consultation with the subscribing firms, are:—

P. C. Allen (Imperial Chemical Industries), T. L. Birrell (Halex), J. L. Daniels (T. H. & J. Daniels), P. A. Delafield (British Resin Products), C. S. Dingley (British Industrial Plastics), H. V. Potter (Bakelite), A. E. Skan (Tufnol).

The trustees have elected P. C. Allen as their chairman; S. P. Thompson, I.C.I. (Plastics Division), has been appointed secretary for 1952; and the Plastics Institute has been asked to act as agent and manager to the trustees to carry on the day-to-day administration of the funds.

Main objects of the fund are to publish monographs on the science and technology of processes used in the plastics industry; to provide training grants to help persons in the industry studying for examinations; to help apprentices in the industry by making grants to cover books, travelling expenses and the like; and to give assistance to educational institutions by providing and maintaining equipment and contributing to the cost of literature and libraries.

### Ten Training Grants

The first specific decision made by the trustees with regard to allocating the fund's resources is the provision of ten training grants to be awarded this year. The Plastics Institute has been asked to invite applications for these grants.

At its next meeting (in May) the trustees of the fund hope to have before them recommendations from the Plastics Institute for further expenditure.

Launched in 1942, the scheme was the first step in laying the foundations of a permanent plan for education in this young industry. The bulk of its funds was in the form of seven-year covenants, and in August last

year it was felt to be essential that the initial scheme, having run its course, should be continued in some form.

Then came the appeal for funds in September, the appointment of trustees this month, and now the first allocation of monies.


While the result of the appeal was fairly satisfactory, only a comparatively small number of firms have subscribed.

A second appeal has therefore been issued, as it is felt that subscription must be on the widest possible basis. The industry must have an adequate number of properly trained technologists, and this is of vital importance to small firms just as it is to large ones.

## U.S. Titanium in 1951

THE Bureau of Mines, U.S. Department of the Interior, has recently reviewed the titanium situation in America during 1951, a year in which several new groups were organised for promoting the production and use of titanium metal. New records were also established for the production of ilmenite, and titanium pigments. Production of titanium sponge in 1951 is estimated at 700 tons from plants operated by Du Pont, Titanium Metals Corporation, N. J. Henderson and the Crane Company. The Henderson plant is the first fully integrated titanium plant with facilities for the chlorination of titanium ore and the recovery of magnesium and chlorine by the electrolysis of magnesium chloride.

The National Research Corporation of Cambridge, Mass., and the Monsanto Chemical Company joined forces during the year to develop an improved process for the production of titanium. Work already in process by National Research was expanded, and additional work was initiated by Monsanto's research department at Dayton, Ohio. The P. R. Mallory-Sharon Titanium Corporation was formed for the development, production and marketing of titanium and its alloys, and a new process for producing titanium metal was under investigation by Horizons, Inc., of Princeton, N.J. This process, under the sponsorship of the Office of Naval Research, had advanced to the pilot plant stage by the end of the year. The Bureau reported that domestic output of ilmenite in 1951 was 488,600 tons.



# The Chemist's Bookshelf

## NEUERE MASSANALYTISCHE METHODEN.

Edited by W. Böttger. Ferdinand Enke Verlag, Stuttgart: 3rd Ed., 1951. Pp. xx + 347. Figs. 30. Paper, Dm. 46. Cloth boards Dm. 49.

This book is Vol. XXXIII of the well-known series, *Die Chemische Analyse*, edited by the late Professor Wilhelm Böttger. British analytical chemists are already familiar with the second edition through its translation by Professor R. E. Oesper, published in 1937. The present edition, whose preparation began as long ago as 1942, will therefore need no recommendation. With regret one notes that the death of Professor Böttger occurred before the final stages of the book were completed; progress through the press was supervised by Dr. Brennecke, one of the contributors.

The present edition is divided into eleven main sections, as follows: I. Elimination of titration errors in acidimetric and alkalimetric titrations; II. Fluorescence indicators in acid-base titrations; III. Titration of weak bases in acetic acid solution; IV. Volumetric estimation of small amounts of water; V. Ceric solutions as volumetric oxidants; VI. Reduction of permanganate to manganate as a volumetric procedure; VII. Iodate and bromate methods. Bromometry. Substitutes for iodometric methods; VIII. Liquid amalgams as reductors in volumetric analysis; IX. Chromous salts as volumetric reductants; X. Oxidation-reduction indicators; XI. Adsorption indicators for precipitation titrations.

There are considerable additions to the contents of the second edition. Sections III, IV and VIII may be regarded as completely new. \*Sections II, VII and XI have been very much expanded, and a substantial amount of new material has been added to the remaining sections.

The book is clearly written and fully documented with numerous references. For a full account of the available information on the fields covered, up to the late 1940's,

it will be found excellent. (A few references dated 1950 are to be found, but the latest year to be reasonably fully represented appears to be 1949).

It may be hoped that this, like the second edition, will achieve translation before long, since much valuable material, hitherto widely scattered, is here brought together, critically examined, and made readily available.—C.L.W.

MEASUREMENT & CONTROL OF TEMPERATURES IN INDUSTRY. By R. Royds, Constable & Co., Ltd., London, 1951. Pp. vii + 260. 25s.

Chapter one of this book covers standard scales of temperature, and is followed by three chapters covering the fundamental methods of measuring temperature. In the first of these, the author groups the liquid in glass, liquid in metal bulbs, vapour in metal bulbs, and bimetallic thermometers under the heading of 'Expansion Thermometers.' Useful ranges of each type are indicated together with precautionary measures to be taken in their use.

Electrical resistance thermometers and thermo-electric pyrometers are covered in detail in Chapter 3. Reference is made to standard equipment available in industry, and details of measuring devices are included. A table is included covering suitable materials for sheaths when subjected to various temperatures and various atmospheric conditions. In this chapter also, the author describes a humidity measuring device based on the use of electrical resistance thermometers.

Radiation pyrometers, optical and photo-electric pyrometers comprise the subject matter of the fourth chapter. The theory of these instruments, sources of error and some of their applications in industry are dealt with in sufficient detail for most purposes.

Galvanometers, indicators, recorders and temperature controllers are covered in

Chapter 5. The section on moving-coil galvanometers is in detail, but the cathode ray tube and recorders are only briefly dealt with in three pages. The majority of the chapter, about 50 pages, deals with regulators or controllers. These are well described, both from the point of view of construction and electrical and electronic circuits, by reference to specific types of instruments manufactured by British or American firms. The theoretical aspects of automatic control are not dealt with.

In Chapter 6, reference is made to calorimetric pyrometers, expansion and contraction pyrometers made from clay and first used by Wedgwood, the use of fusion pyrometers and temperature-indicating points.

The remaining two chapters are of special interest to persons engaged in work on steam

and combustion engines. Chapter 7 deals with the measurement of mean wall temperatures, while the last chapter covers the measurement of fluctuating temperatures. In both cases the author gives many practical details on items such as the mounting of couples, the use of shields and method of fixing wires.

The book concludes with an appendix which deals with the International Temperature Scale of 1948.

Reviewing the work as a whole, there is much useful information on the construction and action of industrial temperature measuring and controlling devices. Applications cited are not numerous, and the general presentation could be improved by introducing more sub-headings into the text.

—E.J.C.

## Qualitative Test for $\text{NH}_4\text{OH}$

A QUALITATIVE test for ammonia in aqueous solution based on the reaction between ammonia and iodine was reported recently in the American *Journal of Chemical Education*, from Oberlin College, Ohio. This reaction is well-known, but has not apparently been used previously for the detection of ammonia. It is, however, more convenient and just as reliable as the usual methods of detection.

One ml. of unknown solution is made neutral to litmus, and two drops excess of 10 per cent NaOH solution added. Any precipitate formed is removed by centrifuging, and the clear solution poured on to about 3 mg. of iodine on a spot plate. A crystal of iodine is pushed to the surface of the liquid with a stirring rod and allowed to slide to the bottom of the depression on the spot plate. A black, finely divided precipitate appears in the wake of the crystal. With small concentrations of ammonia (30 mg./litre), the black precipitate dissolves within a few seconds, but with relatively high concentrations (500 mg./litre) it is more copious and persists much longer.

The test has been used successfully on a variety of solutions and no interference with other ions has yet been discovered. The black precipitate formed in the test is highly explosive when dry, and should be destroyed after the test by adding NaOH or by washing down a drain.

## Improving Vacuum Stopcocks

A METHOD for enlarging the extremely small holes in both the shell and the solid core of vacuum stopcocks is reported in the American *Journal of Chemical Education* from Indiana University. These small holes greatly reduce the pumping speeds in a system, as is well-known, and if several stopcocks are used they may vitiate altogether the work of the operator trying to make the rest of his system of as large a bore as possible.

Solid-core, offset vacuum stopcocks were used, the connecting tubing lying on a straight line through the barrel. A small rat-tail file was moistened with a cutting compound composed of 62 g. of camphor and 90 ml. of turpentine in 45 ml. of ether, and the holes in the barrel carefully enlarged by twisting the file.

In order to enlarge the hole in the solid core, a brass rod from a set of cork borers was tapered at one end for a distance of about 2 in. The rod was placed in a low-speed drill press and the hole was drilled out using a slurry of number 120 carborundum and the above cutting compound as the grinding medium. The stopcock was held in the hand and drilling carried out alternately one side and then the other. As the drilling progressed care was taken that the rod did not bind in the hole. The drilling of each hole needed about 20 minutes.

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# HOME

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## Royal Society Lectures

The Croonian Lecture for 1952 will be delivered on 15 May by Dr. C. F. A. Pantin, F.R.S., Reader in Invertebrate Zoology in the University of Cambridge. The title will be 'The Elementary Nervous System.' The Bakerian Lecture for 1952 will be delivered on 12 June by Professor H. Jeffreys, F.R.S., Plumian Professor of Astronomy in the University of Cambridge. His subject will be 'The Origin of the Solar System.'

## Steel Production Improves

Steel output in March was at an annual rate of 16,648,000 tons compared with 16,281,000 tons in the previous month and 16,546,000 tons in March, 1951. This is the first time this year that the 1951 level has been exceeded. Easter holidays, however, were the cause of last year's comparable figures being relatively low. Pig-iron production in March reached an annual rate of 10,478,000 tons, the highest monthly total so far achieved, compared with 9,572,000 tons a year ago.

## Superannuation Scheme

A non-contributory superannuation scheme for all employees over 21 who have completed two years' service with the company is to be introduced this month by Coalite and Chemical Products, Ltd., of Bolsover and Askern (South Yorks). Commander Colin Buist (chairman) announced the introduction of the scheme at a dinner given by the company to 600 employees. Seventeen employees were presented with gold watches to mark more than 25 years' service with the company.

## Record Production of Glass Containers

The number of glass containers made in Britain in 1951 was 3,600,000,000, according to the Glass Manufacturers' Federation, which is this year celebrating its silver jubilee. In export also, glass, in some form or another, appeared as a component of or package for products valued at £431,474,000. A large proportion of this figure was contributed by chemical, scientific and medical glassware, and scientific instruments.

## Colour in Industry

The exhibition on Colour and Lighting in Industry, postponed from March, will be opened by the Rt. Hon. David Eccles, M.P., Minister of Works, at 12 noon on Friday, 2 May. The exhibition will show colour ranges and demonstrate the principles affecting the use of colour, and also various types of artificial light and their relation to colour.

## Chemical Wages and Hours

Results of an inquiry into the average weekly earnings and working hours of manual workers made in October last are summarised in the *Ministry of Labour Gazette* for March (Vol. 60, No. 3). The number of wage-earners in chemicals and dyes, according to returns received, totalled 109,925 and the average earnings of all workers (including youths and girls) in the last pay-week in October, 1951, was 156s. 2d. For the same group, the average number of hours worked in the same period by all workers was 46.9 at an average hourly rate of 40d.

## Aluminium Scrap Dearer

The price of aluminium scrap was raised as from 1 April in an Order made by the Minister of Supply. Certain additions are permitted by the Order in respect of bagging and special packing and for delivery at consumers' premises. Maximum new prices (with former prices in brackets) are:—New pure (foil scrap for flake powder) £156 (£148), (other than above) £123 (£113); old rolled or extruded pure £112 (£101); clean aluminium alloy £107 (£95); turnings £79 (£69); scrap and alloy scrap other than the four specified above, in proportion; new pure plain aluminium foil stock scrap (as specified in Article 3 of the Order) £149.

## U.K. Copper Price Raised

The price of electrolytic copper was increased by £4 from £227 to £231 a ton delivered consumers' works as from 1 April. Discounts and premiums for special shapes remained unchanged. The higher price was stated to be due to the extent of the overhead charges (including refining) which had to be borne by the Ministry of Supply.

## OVERSEAS

### New Canadian Salt Plant

Negotiations have been completed by International Salt of Canada, Ltd., with the Chesapeake and Ohio Railway for property and rights to mine rock salt at Erieau, Lake Erie, 20 miles south-east of Chatham, Ontario. The agreement was announced by Blake Huffman, Liberal member of Parliament for Kent County, who said that plant valued at \$3,500,000 would be built, and that when completed some 150 men would be employed.

### Japanese Postwar Record

The Japanese Chemical Fibre Industry Association recently reported that the production of rayon and staple fibre yarns during 1951 established a postwar record. Rayon yarn output amounted to 127,900,000 pounds, 24 per cent larger than the 1950 total, and staple fibre production amounted to 230,700,000 pounds, 54 per cent above the 1950 total.

### Unique Project

The Government of Western Australia is now running a unique project at Wundownie, 40 miles from Perth, which combines the operation of wood distillation plants and charcoal-iron production. It includes sawmilling, wood distillation and carbonisation, the refining of pyroligneous liquor, the open cut mining of iron ore, blast furnace smelting and the generation of electric power. Distillation of the native hardwoods produces 500 tons of acetic acid annually, 300 tons of methanol and 1,200 tons of wood tar. The blast furnace has an annual output of 12,000 tons of pig iron.

### Canadian Subsidiary Formed

Formation of a subsidiary company in Canada has been announced by Howard and Sons, Ltd., of Ilford, England, manufacturers of solvents, plasticisers and technical chemicals, which has formerly sold its products through a branch office in Montreal. The new company will be situated in Cornwall, Ontario and will be known as Howard and Sons (Canada), Ltd. Mr. T. H. C. Raikes, of Montreal, has been named president. Plans for a factory are being drawn up and production is scheduled to begin in 1953.

### Chilean Copper Strike Threat

Miners at two of the big properties operated by Anaconda Copper in Chile have voted to come out on strike for increased wages and benefits according to a report by the New York Journal of Commerce. Some 10,000 miners would be affected, it was stated, as well as other workers in the Porterillos mine, with an annual output of 50,000 tons, and the Chuquicamata with an annual output of 172,500 tons.

### Chlorine Explosion at Factory

Four men were killed at a cellulose factory in the Ruhr town of Walsum on 3 April when a tank containing liquid chlorine exploded. The chlorine multiplied rapidly in volume as it became a gas, and for a time it was feared that the whole town was in danger, and bus convoys evacuated people from the factory area. More than 100 were taken to hospital, eight stated to be in a critical condition. The town was reported to be tainted with chlorine 12 hours afterwards and all the available milk supply was distributed as an antidote.

### U.S. Plant for South Africa.

New plant from America which is expected to treble its output of industrial and edible oils was delivered to the Marine Oil Refiners, Ltd., Simonstown, in February. The plant, which will segregate fish oil into drying and edible fractions, is expected to be in operation early in May. About half the production of the refinery is absorbed by the paint and allied industries.

### State Plants Show Profit

It is understood that all industrial alcohol now being made at the five alcohol plants operated by Irish Chemicals, Ltd. (a state-owned company) is being exported to Great Britain at a profit. It is also stated that the contract calls for delivery during 1952 of not less than 1,500,000 gallons valued at about £750,000. The factories were established before the war to support the price of potatoes and critics have always maintained that they were uneconomic because the higher priced alcohol was compulsorily mixed with petrol.



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## PERSONAL

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PROFESSOR JAMES PICKERING KENDALL, professor of chemistry at the University of Edinburgh will be one of the personalities invited by the Senate of Glasgow University to receive the honorary degree of Doctor of Laws at the Commemoration Day graduation ceremony to be held on 18 June.

On Friday evening, 14 March, following a dinner at the Hotel Schenley, Pittsburgh, U.S.A., over 230 members and guests of the Pittsburgh Section of the American Ceramic Society witnessed the presentation of the fifth annual Albert Victor Bleining Memorial Award 'for distinguishing achievement in the field of ceramics' to DR. SAMUEL RAY SCHOLES, Associate Dean and Head of the Department of Glass Technology of the New York State College of Ceramics, Alfred University, Alfred, N.Y.

MR. G. E. SCHARFF, B.A. (Cantab.), F.R.I.C., F.I.R.I., has recently retired from Imperial Chemical Industries, Ltd., after thirty-two years' service in various divisions, and has now become an associate of Dr. G. Lewi in his consulting practice in London.

The Council of The Institute of Fuel have awarded the Melchett Medal for 1952 to PROFESSOR D. T. A. TOWNEND, D.Sc., Ph.D., D.I.C., Hon.M.Inst.Gas E., F.Inst.F., in recognition of his outstanding contributions to the science of combustion, particularly in the field of higher hydrocarbons. Dr. Townend is the Director General of the British Coal Utilisation Research Association. He was formerly Livesey Professor of Coal Gas and Fuel Industries in the University of Leeds, and president of The Institute of Fuel, 1948-50.

Elliott Brothers (London), Ltd., the well-known manufacturers of automatic process control instruments, announce that MR. J. F. COALES has resigned his position as research director of the company.

The Board of Trade has announced with regret the resignation of MR. G. E. HOLDEN, C.B.E., the Dyestuffs Controller. Mr. Holden has held this honorary appointment for the last five years and the President of the Board of Trade has expressed his warm

appreciation of the valuable services he has rendered during his period in office and for many years previously.

It is not intended to replace Mr. Holden in the appointment of Dyestuffs Controller; instead, the Dyestuffs Advisory Committee will be reconstituted and SIR WILLIAM PALMER, K.B.E., C.B., has accepted an invitation from the Board of Trade to become the independent chairman of the new committee.

The Dyestuffs Control will in future be known as the Dyestuffs Office.

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### Obituary

It is with regret that we announce the death of the Independent Chairman of the British Iron & Steel Federation, THE RIGHT HONORABLE SIR ANDREW DUNCAN, G.B.E., on 30 March at the age of 68. Sir Andrew was a director of Imperial Chemical Industries, Ltd., and of the Dunlop Rubber Company, and was a former Minister of Supply and President of the Board of Trade.

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### Erratum

In our report of the annual meeting of the British Chemical Plant Manufacturers' Association in last week's issue we reported that Mr. G. N. Hodson had been elected a vice-chairman of the Association. Unfortunately, Mr. Hodson's company was reported to be Earthenware, Ltd., whereas it should have been Hathenware, Ltd.

### Bituminous Binder Research

Completion of the extension of the laboratories of its bituminous binder research unit in Pretoria was announced in the quarterly review of the Council for Scientific and Industrial Research. For some time now the staff has been engaged in studying the chemical properties of locally made bituminous binders.

### Leather Chemists to Meet in Spain

At the invitation of the Spanish Association of Leather Trades' Chemists, the next biennial conference of Leather Chemists will be held in Barcelona in September next year under the auspices of the International Union of Leather Chemists' Societies.

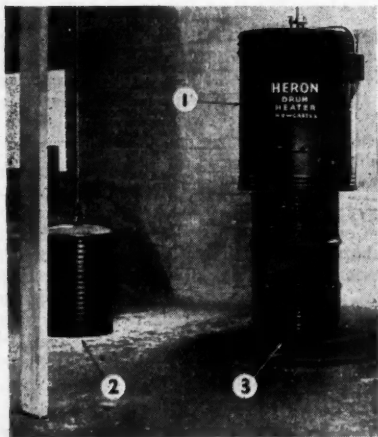


## Publications & Announcements

IN large chemical works and chemical plant it is frequently necessary to employ cranes and sling chains for lifting purposes, and in the majority of cases the importance of not overloading is probably appreciated. Lack of attention to this matter can, however, lead to serious accidents, and a handy reference and pocket safety chart giving the maximum safe working loads on sling chains in good condition has been devised by the Leeds and Bradford Boiler Co., Ltd., Stanningley. On the reverse side of the chart, which is printed on durable plastic, there is a safety chart for lifting steel plates. Originally intended for the firm's own staff, the chart has received the approval of the Royal Society for the Prevention of Accidents and has since been requested by a number of trades. Imperial Chemical Industries Ltd., are said by the company to have ordered a considerable quantity. It is being issued at approximately cost price and may be obtained from the company at 10s. per box of ten, cash with order.

\* \* \*

**PROBLEMS** of releasing solidified material from 45-gallon drums, which have tended



1. The Drum Heater in position to be lowered over a 45 gal. drum of petroleum jelly. 2. Counterbalancing weight for raising and lowering. 3. The drum in position over mixing pan

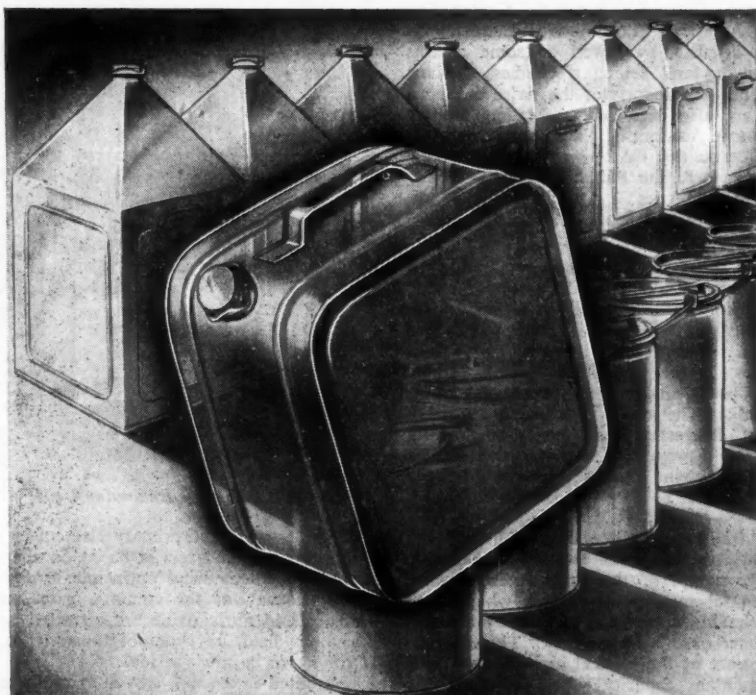
to cause 'bottlenecks' in the oil and paint industries, are claimed to have been overcome by the standard drum heater manufactured by Heron Heating Devices Ltd., Snowdon Street, Newcastle-on-Tyne. The heater consists of an insulated metal casing within which is wound on a former, a special thermal heating wire. It can be used on D.C. or A.C. and is suitable for single or three-phase systems. Any voltage can be used and the standard model consumes approximately 7 kW. Operation is very simple. The drum to be emptied is first placed over the hopper or mixing pan, then the Heron drum heater is lowered over the drum, switched on and left in position for a period of approximately seven minutes. Raising of the temperature expands the sides of the drum, lowers the viscosity of the material in contact with the sides and allows the contents to be released into the mixing pan. Eight drums may be emptied every hour. This method of releasing grease or wax leaves the drum very clean but if washing should be necessary after use the heater can also be used for drying out the moisture. Price of the standard model is approximately £110. The equipment is simple to install and free maintenance for 12 months is guaranteed by the makers.

\* \* \*

**INFLUENCE** of alloy elements on the heat-treatment of grey cast iron is reviewed by M. M. Hallett (chief metallurgist, Sheepbridge Engineering Ltd., Chesterfield), in the March issue of *Alloy Metals Review* (Vol. 8, No. 63), published by High Speed Steel Alloys Ltd., Widnes, Lancashire.

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THE quarterly news letter, *Chemonomics*, published by the consulting chemical engineering firm of R. S. Aries & Associates, New York, contains current news of interest to the chemical process industries with special reference to their economic aspects. Now in its third year of publication, the winter 1951-52 issue contains articles on synthetic cresol derived from toluene; feed additives—an industry with a promising future; continuous charcoal producing units; methods for budgeting research. It also has articles on new products, what is a 'fair' royalty; and Japan—a fertile investment area. A recent suit of interest



## *In the Picture -*

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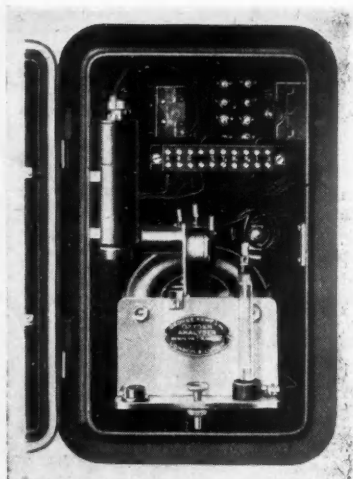
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between Chemical Research Corporation and American Locomotive Company is also discussed as well as some aspects of buying and selling a business. Some activities and reports of the Aries organisation are also described. Readers of THE CHEMICAL AGE can be placed on the permanent free mailing list by writing on company letterheads to the Public Relations Department, R. S. Aries & Associates, 400 Madison Avenue, New York 17, N.Y.

'POWER to Spare,' a magazine published by Oldham and Son, Ltd., has recently published its third number in Volume 5. It contains articles tracing the progress of various industries in Britain from their sources to the present day—shipbuilding, oil, railways, silicones, and a political article on India. The article on silicones describes in an interesting way the fundamental nature and properties of these versatile new materials, giving examples and photographs of their uses.

IN *Sulzer Technical Review* No. 3/1951, the leading article deals with the hydraulic storage pumps of the Etzel Hydro-Electric Power Scheme in Switzerland, which have considerably improved the performance and economy of this large plant. These Sulzer pumps are of the vertical high-lift type with a maximum capacity of 38,165 gals. per min. at a head of 1,568 ft. and an input of 21,450 H.P. and were supplied and installed by a conjoint working enterprise constituted by Messrs. Sulzer Brothers and Messrs. Escher Wyss. The water conditions encountered and the pump-storage plant itself are described and illustrated by drawings, diagrams and photographs. A further long contribution treats of the dynamic behaviour of the regulated system in automatic pressure control installations, and is drawn from research work carried out in the Sulzer thermal laboratory on the pressure control of steam-raising plant.

THE development of a new paramagnetic continuous oxygen recorder for use in industrial processes has been announced by George Kent Ltd., Luton. They claim that the recorder will meet all the usual demands including the determination of the efficiency of the liquid-air process of oxygen manufacture and analysis of waste gases from open-hearth furnaces in the steel industry.



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The design of the primary element of the recorder (the analyser) is based on the so-called 'magnetic wind' principle, owing to the fact that, of the common gases, only oxygen and nitric oxide are attracted by a magnetic field, oxygen more than twice as strongly so as nitric oxide.

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ALTHOUGH the name 'Visco' is well-known in connection with air filters, water coolers and dust-collecting plant, not everyone realises that the firm has a department dealing exclusively with industrial ventilation and air conditioning. A new pamphlet (No. 523) gives illustrations and brief particulars of a few of the industrial installations that the firm has carried out in various plants. The plants dealt with include those at May & Baker, Glaxo Laboratories, Walton & Brown, the United Coke & Chemical Company and G.E.C.

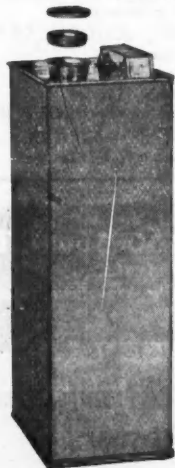
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## Next Week's Events

### MONDAY, 14 APRIL

#### Incorporated Plant Engineers

Dundee: Mathers Hotel, 7.30 p.m. Film on the manufacture of plate glass by Pilkington Brothers, Ltd.

### TUESDAY, 15 APRIL

#### Incorporated Plant Engineers

Glasgow: 351 Sauchiehall Street, 7 p.m. T. C. Crawhall (assistant director, Mechanical Research Organisation): 'Development of the New Mechanical Research Laboratory at East Kilbride.'

### WEDNESDAY, 16 APRIL

#### Institute of Fuel

Manchester: Engineers' Club, Albert Square, 2 p.m. Annual general meeting; 2.30 p.m. Professor R. J. Sarjant and W. Hulse: 'Thermal Factors in Furnace Design.'

#### Institute of Welding

Manchester: Reynolds Hall, College of Technology, 7 p.m. Annual general meeting, followed by film display.

#### Institution of Engineering Inspection

Manchester: Engineers' Club, Albert Square, 7.30 p.m. Ian Scott (Oil Well Engineering Co., Ltd.): 'Some Features of the Oil Industry.'

#### Incorporated Plant Engineers

Bristol: Grand Hotel, 7.15 p.m. C. Gotsell: 'Industrial Fire Protection.'

### THURSDAY, 17 APRIL

#### Society of Chemical Industry

London: Royal College of Science, Imperial Institute Road, South Kensington, 10.30 a.m. and 2.15 p.m. Joint meeting of the Microbiology Group with the British Society of Soil Science on 'Soil Nitrogen Status.' Various speakers and discussion.

#### Royal Institute of Chemistry

Luton: The Town Hall, 7.30 p.m., with the Luton Scientific Association. Dr. F. Sherwood Taylor (director, Science Museum): 'Some Aspects of the History of Science.'

#### Pharmaceutical Society

Manchester: Deansgate, Houldsworth Hall, 7.30 p.m. Junior branch, annual general meeting.

### FRIDAY, 18 APRIL

#### The Chemical Society

St. Andrews: United College, 5.15 p.m. Joint meeting with the Royal Institute of Chemistry. Professor E. R. H. Jones: 'Polyacetylenes.'

#### Institute of Metals

Sheffield: University, St. George's Square, 6.30 p.m. Joint meeting with the Institute of Metal Finishing. Dr. U. R. Evans: 'Corrosion of Iron and its Prevention by Deposits of Non-Ferrous Metals.'

#### Liverpool Metallurgical Society

Liverpool: Electricity Service Centre, Whitechapel, 7 p.m. Annual general meeting and presentation of prize-winning student papers.

#### Institution of Electronics

Manchester: Reynolds Hall, College of Technology, 7 p.m. D. Latham (W. Edward and Co. (London), Ltd.): 'Vacuum Pumps and Techniques.'

#### Society of Dyers and Colourists

Manchester: Gas showrooms, Town Hall Extension, 6.30 p.m. Annual general meeting.

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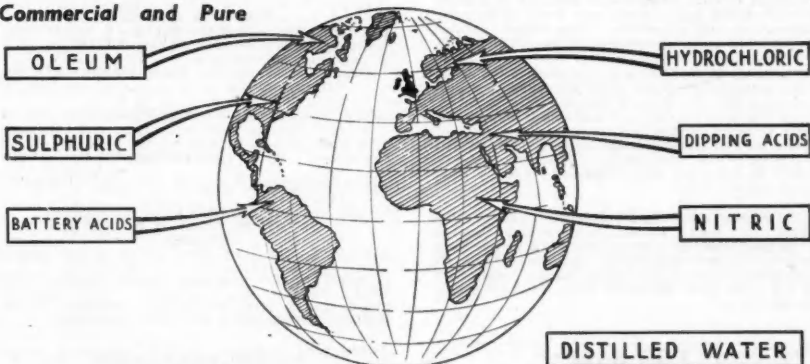
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# CLASSIFIED ADVERTISEMENTS

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### HER MAJESTY'S COLONIAL SERVICE

APPLICATIONS are invited for the following post:—  
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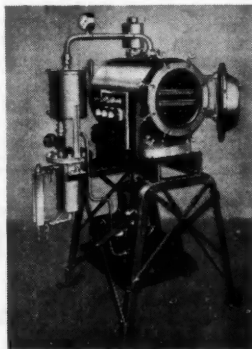
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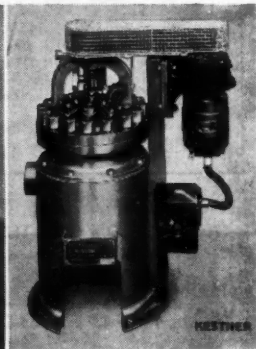


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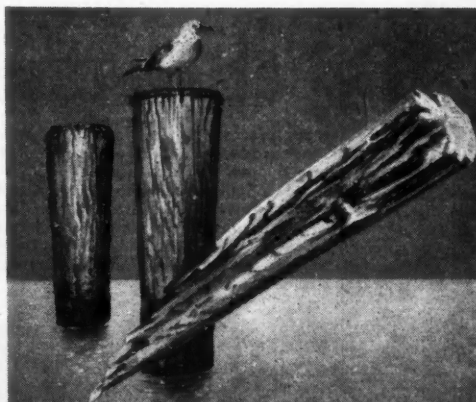
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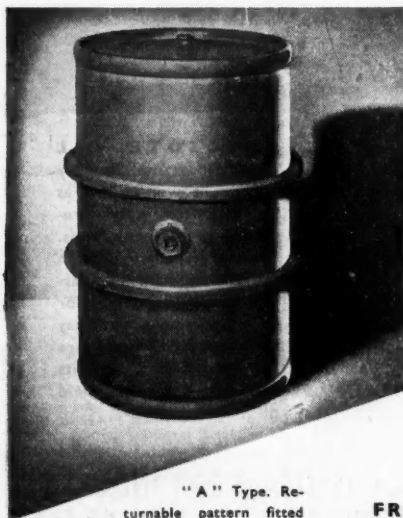
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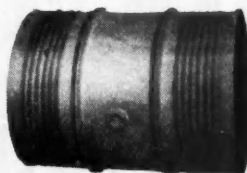
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